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AFRPL-TR-66-318

(Unclassified Title)

CHLORINE PENTAFLUORIDE HANDBOOK

Special Technical Report

By
Chemist, Section
Research Division
Rockwell
A Division of North American Aviation, Inc.
Canoga Park, California

TECHNICAL REPORT AFRPL-TR-66-318

November 1966

Group 4
Downgraded at 3-Year Intervals
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FOREWORD

This interim propellant handbook is submitted as a special technical report under Rocketdyne G.O. 7170 in compliance with Contract F04611-67-C-0006, Part I, Subline Item IAA. The effort under this contract is being sponsored by the Air Force Rocket Propulsion Laboratory, Research and Technology Division, Air Force Systems Command, USAF, Edwards, California, with 1/Lt. Ralph Fagnoli acting as the Air Force Project Officer.

The compilation, analysis, and publication of the data and information contained herein is part of a 9-month program to provide a comprehensive and systemized compilation of the properties, handling procedures, and design criteria for chlorine trifluoride and chlorine pentafluoride. This interim manual on chlorine pentafluoride is designed to meet immediate industry and government requirements until the publication of the final handbook. The final handbook, which is scheduled for distribution by 31 August 1967, will expand the ClF₅ data and information contained in this handbook to include detailed design criteria.

This program is being conducted in the Chemical Research Section of the Rocketdyne Research Division, with Dr. J. Silverman serving as Program Manager and Mr. M. T. Constantine serving as Responsible Project Scientist.

The following technical personnel contributed to compilation and analysis of the data and information contained in this handbook: M. M. Williams, K. J. Youel, D. Pilipovich, C. J. Rozas, and E. J. Walter.

This handbook has been assigned the Rocketdyne identification number R-6803.

Publication of this report does not constitute Air Force approval of the report's findings or conclusions. It is published only for the exchange and stimulation of ideas.

W. H. EBELKE, Colonel, USAF
Chief, Propellant Division

ACKNOWLEDGEMENT

This interim handbook includes data and information generated through experimental and analytical studies conducted at many different organizations. Although every effort has been made to reference these studies, a great number of individuals responsible for these data have been neglected. These individual contributions as well as the contributions of the industry and government organizations are gratefully acknowledged.

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ABSTRACT

This interim handbook is a compilation of the engineering properties and handling characteristics of chlorine pentafluoride. The handbook includes data and information on chlorine pentafluoride physico-chemical properties, mixtures, materials compatibility, materials preparation, toxicity, fire and explosion hazards, hazard prevention and control, safety equipment, decontamination, production, propellant analysis, transportation, storage, and handling.

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SECTION 1: INTRODUCTION

1.1 GENERAL

During the course of an Office of Naval Research contract on basic fluorine chemistry studies (Ref. 1) in 1960, a new fluorine-containing compound was synthesized by investigators at the Rocketdyne Division of North American Aviation, Inc. Sufficient quantities of the compound were not readily available at that time to identify and characterize the material unambiguously. The unknown material was, therefore, designated as Compound A. In 1962, under Air Force Rocket Propulsion Laboratory sponsorship (Ref. 2), Compound A was definitely identified as chlorine pentafluoride (ClF_5) by the same investigators.

Preliminary characterization of chlorine pentafluoride, which bore a strong chemical and physical resemblance to its lower interhalogen homologue, chlorine trifluoride, indicated that a significant new addition had been made to the small family of dense, high-performance, stable liquid rocket oxidizers. Because of the potential usefulness and near-term application of this compound to rocket propulsion systems, the Department of Defense accordingly supported continuing investigations with chlorine pentafluoride to complete the chemical and physical characterization of the material and establish its utility in such systems.

This handbook is a current summary of the engineering properties of chlorine pentafluoride. The material presented is that which has evolved from various engineering characterization studies, applicable handling experience, and the chemical similarity of the compound to chlorine trifluoride. The handbook is intended as a guide for those involved in the handling and application of this oxidizer.

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1.2 HANDBOOK FORMAT

The material contained in this handbook has been organized into seven sections. These are:

- Section 1: Introduction
- Section 2: Physico-Chemical Properties
- Section 3: Materials of Construction
- Section 4: Hazards
- Section 5: Safety Criteria
- Section 6: Logistics
- Section 7: References

Each section is subdivided further, to permit the user of the handbook to obtain specific information expeditiously. The material is arranged in such a manner as to permit convenient updating of various sections as data are generated from additional studies in these areas.

The interest of each individual user may be limited to specific aspects of the subject material; however, it is recommended that personnel involved in ClF_5 handling be thoroughly familiar with all of the material contained in this report. Although every effort has been made to provide presently available information on ClF_5 in sufficient detail for most of the potential users of the handbook, size limitations of the handbook obviously preclude inclusion of every conceivable detail. Thus, for those users who desire additional details on specific items, consultation of the referenced publications is recommended.

Wherever possible, the data and information referenced are from final reports. This was done to eliminate confusion in efforts where progress reports included incomplete experimentation and/or

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analysis of the data. In those efforts where a final report has not been issued, the data were taken from the latest progress report containing pertinent results.

Because the major portion of this handbook is related to areas of engineering interests, all of the data are presented in engineering terminology (i.e., English units). However, as a convenience to all of the users, data in certain sections (notably, the physical properties section) of the handbook are presented in both metric and English units. Where data are presented in both units, the attendant discussion indicates the units of the referenced work.

As a further convenience to the user, physical constants and conversion factors are presented in Tables 1 through 3 to enable the user to convert the values to his particular needs. Also, because these constants are presented to the known degree of significance, they can be rounded to fit particular needs.

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TABLE 1

PHYSICAL CONSTANTS

<u>Unit</u>	<u>Remarks</u>	<u>Value</u>
g_c	Standard gravitational acceleration	32.1740 ft/sec ² 980.665 cm/sec ²
1 atm	Standard atmosphere	1,013,250 dynes/sq cm
1 mm Hg	Standard millimeter Hg	1333.2237 dynes/sq cm
1 cal	Thermochemical calorie	4.1840 abs joules 41.2929 ± 0.0020 cu cm-atm
1 cal (I. T.)	International Steam Tables calorie	1.000654 thermochemical calories
T_0 C	Ice Point	491.6880 ± 0.018 R 273.160 ± 0.010 K
$(PV)_{0\text{ C}}^{P=0} = (RT)_{0\text{ C}}$	Pressure-Volume product for ideal gas at 0 C	22,414.6 ± 0.4 cu cm-atm/g mole 2271.16 ± 0.04 abs joules/g mole
R	Molar gas constant	8.31439 ± 0.00034 abs joules/K-g mole 1.98719 ± 0.00013 cal/K-g mole 82.0567 ± 0.0034 cu cm-atm/K-g mole 59.47 cu ft-atm/R-lb mole 10.73 cu ft-psia/R-lb mole
1 Btu		1055.040 abs joules 252.161 thermochemical calories 251.996 I. T. calories
1 in.	United States unit	2.54000508 cm
1 ft	United States unit	30.4800610 cm
1 lb	Avoirdupois	453.5924277 g
1 gal	United States unit	0.133680555 cu ft 3785.43449 cu cm

Note: Compiled by Rossini, F. D. et al, American Petroleum Institute Research Project 44, U.S. Department of Commerce, Natl. Bur. Standards, Circular 461, U.S. Government Printing Office, Washington, D. C. 1947.

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TABLE 2

CONVERSION FACTORS

Temperature

$$C + 273.16 = K$$

$$F + 459.58 = R$$

$$(C \times 1.8) + 32 = F$$

$$(F - 32)/1.8 = C$$

$$K(1.8) = R$$

Pressure

$$\text{atm} \times 14.69618 = \text{psi}$$

$$\text{mm Hg} \times 0.00131579 = \text{atm}$$

$$\text{mm Hg} \times 0.019337 = \text{psi}$$

$$\text{g/sq cm} \times 0.00096784 = \text{atm}$$

$$\text{g/sq cm} \times 0.0142234 = \text{psi}$$

$$\text{bars} \times 0.98692 = \text{atm}$$

$$\text{bars} \times 14.504 = \text{psi}$$

$$\text{megabaryes} \times 1 = \text{bars}$$

Mass

$$\text{grams (mass)} \times 0.002204622 = \text{pounds (mass)}$$

Length

$$\text{centimeters} \times 0.393700 = \text{inches}$$

$$\text{centimeters} \times 0.032808 = \text{feet}$$

Area

$$\text{square centimeters} \times 0.15500 = \text{square inches}$$

$$\text{square centimeters} \times 0.0010764 = \text{square feet}$$

$$\text{square feet} \times 144 = \text{square inches}$$

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TABLE 2
(Continued)

Volume

cubic centimeters x 0.061023 = cubic inches
cubic centimeters x 3.531445×10^{-5} = cubic feet
cubic inches (U.S.) x 5.78704×10^{-4} = cubic feet

Time

seconds/60 = minutes
seconds/3600 = hours
seconds/86,400 = days

Force

dynes x 0.00101972 = grams (force)
grams (force) x 0.00220462 = pounds (force)

Density and Specific Volume

(g/cu cm) x 62.43 = lb/cu ft
(cu cm/g) x 0.016018 = cu ft/lb

Surface Tension

(dynes/cm) x 6.8523×10^{-5} = lbf/ft

Thermodynamic Properties

(cal/g mole) x 1.8 = Btu/lb mole
(cal/g mole-R) x 1 = Btu/lb mole - R
(Btu/lb mole)/mol. wt = Btu/lb
(Btu/lb mole-R)/mol. wt = Btu/lb - R

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TABLE 2

(Concluded)

Viscosity

centipoises $\times 0.672 \times 10^{-3} = \text{lb/ft-sec}$

centistokes $\times 1.076 \times 10^{-5} = \text{sq ft/sec}$

(kinematic viscosity) \times (density) = (absolute) viscosity

Thermal Conductivity

(cal/cm-sec-C) $\times 241.8588 = \text{Btu/ft-hr-F}$

Velocity of Sound

(m/sec) $\times 3.28083 = \text{ft/sec}$

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TABLE 3
TEMPERATURE CONVERSION

-559 to 0			0 to 50			50 to 100			100 to 500			500 to 1000			1000 to 1500			1500 to 2000		
C	F	R	C	F	R	C	F	R	C	F	R	C	F	R	C	F	R	C	F	R
-273	-459		-17.8	0	32.0	10.0	50	122.0	38	100	212	260	500	932	538	1000	1832	816	1500	2732
-268	-450		-17.2	1	33.8	10.6	51	123.8	38	110	230	266	510	950	543	1019	1850	821	1510	2750
-262	-440		-16.7	2	35.6	11.1	52	125.6	38	120	248	271	520	968	549	1029	1868	827	1520	2768
-257	-430		-16.1	3	37.5	11.7	53	127.5	38	130	266	277	530	986	555	1039	1886	832	1530	2786
-251	-420		-15.6	4	39.2	12.2	54	129.2	38	140	285	282	540	1005	560	1050	1905	838	1540	2805
-246	-410		-15.0	5	41.0	12.8	55	131.0	38	150	302	288	550	1022	566	1060	1922	843	1550	2822
-240	-400		-14.5	6	42.8	13.3	56	132.8	38	160	320	293	560	1040	571	1070	1940	849	1560	2840
-234	-390		-13.9	7	44.6	13.9	57	134.6	38	170	338	299	570	1058	577	1080	1958	855	1570	2858
-229	-380		-13.3	8	46.5	14.5	58	136.5	38	180	356	304	580	1076	582	1090	1976	860	1580	2876
-223	-370		-12.8	9	48.2	15.0	59	138.2	38	190	375	310	590	1094	588	1100	1994	866	1590	2894
-218	-360		-12.2	10	50.0	15.6	60	140.0	38	200	392	316	600	1112	593	1110	2012	871	1600	2912
-212	-350		-11.7	11	51.8	16.1	61	141.8	38	210	410	321	610	1130	599	1120	2030	877	1610	2930
-207	-340		-11.1	12	53.6	16.7	62	143.6	38	220	428	327	620	1148	605	1130	2048	882	1620	2948
-201	-330		-10.6	13	55.5	17.2	63	145.5	38	230	446	332	630	1166	610	1140	2066	888	1630	2966
-196	-320		-10.0	14	57.2	17.8	64	147.2	38	240	464	338	640	1184	616	1150	2084	893	1640	2984
-190	-310		-9.5	15	59.0	18.3	65	149.0	38	250	482	343	650	1202	621	1160	2102	899	1650	3002
-184	-300		-8.9	16	60.8	18.9	66	150.8	38	260	500	349	660	1220	627	1170	2120	905	1660	3020
-179	-290		-8.3	17	62.6	19.5	67	152.6	38	270	518	355	670	1238	632	1180	2138	910	1670	3038
-173	-280		-7.7	18	64.5	20.0	68	154.5	38	280	536	360	680	1256	638	1190	2156	916	1680	3056
-169	-271	-459	-7.2	19	66.2	20.6	69	156.2	38	290	554	366	690	1274	643	1200	2174	921	1690	3074
-164	-270	-454	-6.6	20	68.0	21.1	70	158.0	38	300	572	371	700	1292	649	1210	2192	927	1700	3092
-162	-260	-456	-6.1	21	69.8	21.7	71	159.8	38	310	590	377	710	1310	655	1220	2210	932	1710	3110
-157	-250	-468	-5.5	22	71.6	22.2	72	161.6	38	320	608	382	720	1328	660	1230	2228	938	1720	3128
-151	-240	-480	-5.0	23	73.5	22.8	73	163.5	38	330	626	388	730	1346	666	1240	2246	943	1730	3146
-146	-230	-482	-4.4	24	75.2	23.3	74	165.2	38	340	644	393	740	1364	671	1250	2264	949	1740	3164
-140	-220	-484	-3.8	25	77.0	23.9	75	167.0	38	350	662	399	750	1382	677	1260	2282	955	1750	3182
-134	-210	-486	-3.3	26	78.8	24.5	76	168.8	38	360	680	404	760	1400	682	1270	2300	960	1760	3200
-129	-200	-488	-2.7	27	80.6	25.0	77	170.6	38	370	698	410	770	1418	688	1280	2318	966	1770	3218
-123	-190	-490	-2.2	28	82.5	25.6	78	172.5	38	380	716	416	780	1436	693	1290	2336	971	1780	3236
-118	-180	-492	-1.6	29	84.2	26.1	79	174.2	38	390	734	421	790	1454	699	1300	2354	977	1790	3254
-112	-170	-494	-1.1	30	86.0	26.7	80	176.0	38	400	752	427	800	1472	704	1310	2372	982	1800	3272
-107	-160	-496	-0.5	31	87.8	27.2	81	177.8	38	410	770	432	810	1490	710	1320	2390	988	1810	3290
-101	-150	-498	0	32	89.6	27.8	82	179.6	38	420	788	438	820	1508	716	1330	2408	993	1820	3308
-95.6	-140	-500	0.5	33	91.5	28.3	83	181.5	38	430	806	443	830	1526	721	1340	2426	999	1830	3326
-90.0	-130	-502	1.1	34	93.2	28.9	84	183.2	38	440	824	449	840	1544	727	1350	2444	1005	1840	3344
-84.4	-120	-504	1.6	35	95.0	29.5	85	185.0	38	450	842	455	850	1562	732	1360	2462	1010	1850	3362
-78.9	-110	-506	2.2	36	96.8	30.0	86	186.8	38	460	860	460	860	1580	738	1370	2480	1016	1860	3380
-73.3	-100	-508	2.7	37	98.6	30.6	87	188.6	38	470	878	466	870	1598	743	1380	2498	1021	1870	3398
-67.8	-90	-510	3.3	38	100.4	31.1	88	190.4	38	480	896	471	880	1616	749	1390	2516	1027	1880	3416
-62.2	-80	-512	3.8	39	102.1	31.7	89	192.2	38	490	914	477	890	1634	755	1400	2534	1032	1890	3434
-56.7	-70	-514	4.4	40	104.0	32.2	90	194.0	38	500	932	482	900	1652	760	1410	2552	1038	1900	3452
-51.1	-60	-516	5.0	41	105.8	32.8	91	195.8	38	510	950	488	910	1670	766	1420	2570	1043	1910	3470
-45.6	-50	-518	5.5	42	107.6	33.3	92	197.6	38	520	968	493	920	1688	771	1430	2588	1049	1920	3488
-40.0	-40	-520	6.1	43	109.5	33.9	93	199.5	38	530	986	499	930	1706	777	1440	2606	1055	1930	3506
-34.4	-30	-522	6.6	44	111.2	34.4	94	201.2	38	540	1004	505	940	1724	782	1450	2624	1060	1940	3524
-28.9	-20	-524	7.2	45	113.0	35.0	95	203.0	38	550	1022	510	950	1742	788	1460	2642	1066	1950	3542
-23.3	-10	-526	7.8	46	114.8	35.6	96	204.8	38	560	1040	516	960	1760	793	1470	2660	1071	1960	3560
-17.8	0	-528	8.3	47	116.6	36.1	97	206.6	38	570	1058	521	970	1778	799	1480	2678	1077	1970	3578
			8.9	48	118.4	36.7	98	208.4	38	580	1076	527	980	1796	805	1490	2696	1082	1980	3596
			9.5	49	120.2	37.2	99	210.2	38	590	1094	532	990	1814	810	1500	2714	1088	1990	3614
			10.0	50	122.0	37.8	100	212.0	38	600	1112	538	1000	1832				1093	2000	3632

Interpolation Factors

C	F	R
0.56	1	1.8
1.11	2	3.6
1.67	3	5.4
2.22	4	7.2
2.78	5	9.0
3.33	6	10.8
3.89	7	12.6
4.44	8	14.4
5.00	9	16.2
5.56	10	18.0

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SECTION 2: PHYSICO-CHEMICAL PROPERTIES

2.1 GENERAL DESCRIPTION

Chlorine pentafluoride (CPF, Compound A, Fluoridyne) is a halogen fluoride having the empirical formula ClF_5 . The color of the propellant is white in the solid state, water-white in the liquid state, and colorless in the gaseous state. The odor has been described as both sweet and pungent, similar to fluorine, chlorine trifluoride, chlorine, or mustard. It is insensitive to mechanical shock, nonflammable in air, and exhibits excellent thermal stability over its entire liquid range.

Chlorine pentafluoride can be a hazardous propellant because of its toxicity and reactivity. The greatest hazard of the propellant lies in its reactivity; it reacts with the vast majority of organic and inorganic compounds and, under proper conditions, with most common metals.

Chlorine pentafluoride boils at approximately 7 F and has a vapor pressure of approximately 49 psia at 68 F. As a result, refrigeration is not required to keep the propellant in the liquid state in conventional moderate-pressure vessels. When stored and/or transferred in clean, dry, compatible systems, by properly trained personnel, chlorine pentafluoride does not present a serious storage or handling problem.

2.2 PHYSICAL PROPERTIES

Selected physical properties of chlorine pentafluoride have been defined in a number of experimental and analytical investigations. Nominal values for various phase, thermodynamic, transport, and electromagnetic properties, that have been recommended as the most representative of the existing data, are summarized in

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Table 4. The recommended data were selected from those studies in which the sample composition was ≥ 98 w/o ClF_5 . All of the presented data are direct experimental determinations or are derived from curve-fits of the experimental data, except for those data referenced with an asterisk; the starred data are calculations made during the referenced work based on standard analytical correlations and physical relationships. The absence of data on a particular property is denoted by blank spaces.

Properties, for which property-temperature relationships have been established, are noted in Table 4 with a figure number. The values listed at a nominal temperature were established from their respective property-temperature plots. These graphical illustrations (Fig. 1 through 10) represent either curve-fits of the best available experimental data or analytical estimations of the property; curve-fits of experimental data are noted with a solid line, while a dashed line designates calculated data. Equations representing the curve-fits over the noted temperature ranges are included.

The origin of the selected data is referenced in Table 4 and in each individual figure. A brief discussion of the available data for each property is presented in the following paragraphs.

2.2.1 General Identification

The physical classifications under general identification are those properties that are used to identify a particular substance and its physical state.

2.2.1.1 Molecular Weight. The molecular weight of chlorine pentafluoride was experimentally determined by vapor density measurements as a means of preliminary identification of ClF_5 (Ref. 3). The resulting experimental value of 128 compared favorably to that value (130.5) calculated from the International Atomic Weights in conjunction with the molecular formula.

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2.2.1.2 Freezing Point. Experimental measurements on a ClF_5 sample of >99 w/o purity (Ref. 3) established a freezing point of -103 ± 4 C. A freezing point of -96 C was measured on a ClF_5 sample of ~96 w/o purity (Ref. 4).

2.2.1.3 Normal Boiling Point. The normal boiling point (equilibrium vapor pressure of 760 mm Hg) of an "ultrapure" (>99.9 w/o ClF_5) sample of ClF_5 was experimentally measured as -13.7 C (Ref. 5). Boiling points of -13.1 C (Ref. 6), -13.6 (Ref. 3), and -18.1 (Ref. 4), were calculated from equations representing experimental vapor pressure measurements on less pure ClF_5 samples.

2.2.1.4 Critical Properties. The ClF_5 critical point was experimentally determined as 143 ± 0.5 C, 771 psia, and 0.565 gm/cc (Ref. 6).

2.2.2 Phase Properties

Those properties of ClF_5 , which are associated with one particular phase (either solid, liquid, or gas), have been grouped as phase properties.

2.2.2.1 Density. The saturated liquid density of ClF_5 has been established from -80 C to the critical point as a result of two experimental efforts (Ref. 3 and 6). These data, which are shown in Fig. 1 and 1A, were curve-fitted from -80 C (-112 F) to 99 C (210 F) with the following equations:

$$\begin{aligned} \rho(\text{gm/cc}) = & 3.553 - 1.396 \times 10^{-2} T_{(\text{K})} + \\ & 4.565 \times 10^{-5} T_{(\text{K})}^2 - 6.3111 \times 10^{-8} T_{(\text{K})}^3 \end{aligned}$$

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and

$$\rho(\text{lb/ft}^3) = 221.8 - 48.42 \times 10^{-4} T_{(R)} + \\ 87.96 \times 10^{-5} T_{(R)}^2 - 67.56 \times 10^{-8} T_{(R)}^3$$

Liquid density measurements were also conducted from 0 to 70.5 C on a ClF_5 sample of unreported purity (Ref. 7), and from -77 to 27.5 C on a ClF_5 sample of ~96 w/o purity (Ref. 4). Density equations representing these data are:

$$\rho(\text{gm/cc}) = 1.832 - 3.71 \times 10^{-3} T_{(R)}$$

and

$$\rho(\text{gm/cc}) = 1.776 - 31 \times 10^{-4} T_{(C)}$$

No density data have been reported for the solid and gas phases of ClF_5 .

2.2.2.2 Vapor Pressure. Vapor pressure measurements on ClF_5 have been conducted by a number of investigators (Ref. 3, 4 and 6 through 8). The recommended data, which are shown in Fig. 2 and 2A are a correlation (Ref. 6) of the results from two (Ref. 3 and 6) of these studies. Equations representing these data from -80 C (-112 F) to the critical point are

$$\log P_{(\text{atm})} = 4.6029 - 1197/T_{(K)}$$

and

$$\log P_{(\text{psia})} = 5.7701 - 2154.6/T_{(R)}.$$

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With the exception of measurements conducted on ClF_5 of ~96 w/o purity (Ref. 4), all of the results from the other studies were in good agreement with the selected data.

2.2.2.3 Surface Tension. The surface tension of ClF_5 , which was experimentally determined over a temperature range of -50 to 25 C (Ref. 6), is shown in Fig. 3 and 3A. The equations representing these data are

$$\gamma_{\text{(dynes/cm)}} = 57.949 - 0.14463T_{\text{(K)}}$$

and

$$\gamma_{\text{(lb/ft)}} = 3.9708 \times 10^{-3} - 0.55058 \times 10^{-5}T_{\text{(R)}}.$$

2.2.2.4 Coefficient of Thermal Expansion. No data are available on the coefficient of thermal expansion of ClF_5 .

2.2.2.5 Coefficient of Compressibility. The adiabatic compressibility of liquid ClF_5 was calculated from experimental sonic velocity and density data on the saturated liquid (Ref. 5). Graphical representations of these data, shown in Fig. 4 and 4A, can be described by the following equations:

$$\begin{aligned} \beta_{\text{(atm}^{-1}\text{)}} &= 1.1565 \times 10^{-4} + 1.3942 \times 10^{-6}T_{\text{(C)}} + \\ &1.2708 \times 10^{-8}T_{\text{(C)}}^2 + 1.4680 \times 10^{-10}T_{\text{(C)}}^3 + \\ &9.6855 \times 10^{-13}T_{\text{(C)}}^4 \end{aligned}$$

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and

$$\begin{aligned}\beta \text{ (psi}^{-1}\text{)} &= 6.4065 \times 10^{-6} + 4.0065 \times 10^{-8} T_{(F)} + \\ &1.4103 \times 10^{-10} T_{(F)}^2 + 9.0915 \times 10^{-13} T_{(F)}^3 + \\ &6.2782 \times 10^{-15} T_{(F)}^4\end{aligned}$$

An empirical relationship based on a common molecular parameter was used to calculate the isothermal compressibility (Ref. 6). These data were corrected (Ref. 9) and expanded, and the results are presented in Fig. 5 and 5A.

2.2.2.6 Inert Gas Solubility. Current experimental measurements on gaseous N_2 solubility in ClF_5 have resulted in an average differential solubility value of $\sim 3.0 \times 10^{-5}$ lb N_2 /lb ClF_5 -psi at 120 F (Ref. 10). These measurements are being extended to other temperatures and to the determination of helium solubility in ClF_5 .

2.2.3 Thermodynamic Properties

The properties of ClF_5 which define energy changes in the physical transitions through the various solid, liquid, and gas states, as well as in chemical changes, are classified under thermodynamic properties.

2.2.3.1 Heat of Formation. The heat of formation of liquid ClF_5 has been established by the Joint Army-Navy-Air Force (JANAF) Thermochemical Panel as -60.5 ± 6.0 kcal/mole at 298 K (Ref. 11). This value represents an average of ΔH_f values resulting from experimental

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measurements of the heats of reaction (Ref. 3) of ClF_5 with gaseous H_2 and gaseous NH_3 and the heat of hydrolysis in the presence of hydrazine hydrochloride (Ref. 12).

2.2.3.2 Heat of Fusion. There are no available data on the heat of fusion of ClF_5 .

2.2.3.3 Heat of Vaporization. A ClF_5 heat of vaporization value of 5.313 kcal/mole was calculated from the Clapeyron equation using the selected vapor pressure data (Ref. 6).

2.2.3.4 Heat Capacity. The heat capacity of saturated liquid ClF_5 has been experimentally determined during two different studies over temperature ranges of -50 to 50 C (Ref. 6) and -72.9 to -31.0 C (Ref. 13). The data from the first study (Ref. 6) was analytically re-evaluated and corrected during a subsequent study (Ref. 14). Although there are unexplainable discrepancies between the two different sets of data, the data over the larger temperature range were arbitrarily selected as the recommended values. These data, which are shown in Fig. 6 and 6A, are defined with the following equations (Ref. 14):

$$C_p(\text{cal/gm-K}) = 1.0847 - 0.9522 \times 10^{-2}T(\text{K}) + \\ 0.3595 \times 10^{-4}T(\text{K})^2 - 0.4309 \times 10^{-7}T(\text{K})^3$$

and

$$C_p(\text{Btu/lb-R}) = 1.0847 - 0.5290 \times 10^{-2}T(\text{R}) + \\ 0.1110 \times 10^{-4}T(\text{R})^2 - 0.7389 \times 10^{-8}T(\text{R})^3$$

No data have been generated on the heat capacity of solid or gaseous ClF_5 .

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2.2.4 Transport Properties

All properties of ClF_5 that involve the transfer of mass or energy at the molecular level are presented in the following paragraphs.

2.2.4.1 Viscosity. The recommended viscosity data for saturated liquid ClF_5 are a correlation (Ref. 14) of experimental data resulting from two different studies (Ref. 6 and 13) with overlapping temperature ranges. The equations, which describe the data shown in Fig. 7 and 7A, are:

$$\log \eta_{(c_p)} = -1.62875 + 335.636/T(K)$$

$$\log \eta_{(lb/ft-sec)} = -4.80138 + 604.145/T(R)$$

Although no viscosity data are available on gaseous ClF_5 , additional efforts are in progress (Ref. 10) to expand the liquid data to higher temperatures under both saturated and pressurized conditions.

2.2.4.2 Thermal Conductivity. Although no experimental data are available, the thermal conductivity of liquid ClF_5 has been estimated using a technique described by Weber (Ref. 15). This technique relates thermal conductivity to density and heat capacity through the following equation:

$$K = 3.59 \times 10^{-3} c_p \rho \left(\frac{\rho}{M}\right)^{1/3}$$

where

- K = thermal conductivity
- c_p = heat capacity
- ρ = density
- M = molecular weight

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Using experimental density data (Ref. 6) and heat capacity data (Ref. 14), the thermal conductivity of ClF_5 was calculated from -50 (-58 F) to 50 C (122 F) and is graphically illustrated in Fig. 8 and 8A .

2.2.4.3 Velocity of Sound. Experimental sonic velocity measurements have been conducted in saturated liquid ClF_5 from -77.1 to 68.7 C (Ref. 10). A curve-fit of the data, shown in Fig. 9 and 9A , resulted in the following equations:

$$c(\text{m/sec}) = 1755 - 4.074T(\text{K}) + 5.936 \times 10^{-4}T(\text{K})^2$$

and

$$c(\text{ft/sec}) = 5758 - 7.426T(\text{R}) + 6.011 \times 10^{-4}T(\text{R})^2$$

Sonic velocity measurements were also conducted in liquid ClF_5 under total pressurizations (with gaseous nitrogen) of 500 and 1000 psia (Ref. 10) over temperature ranges of -73.5 to 69.2 C and -27.0 to 16.8 C, respectively. These data, in which certain anomalies were apparent (Ref. 10), were curve fitted with the following equations:

At 500-psia pressurization (199.7 to 342.4 K)

$$c(\text{m/sec}) = 1967 - 5.869T(\text{K}) + 4.128 \times 10^{-3}T(\text{K})^2$$

while at 1000-psia pressurization (246.2 to 290.0 K)

$$c(\text{m/sec}) = 1530 - 3.133T(\text{K})$$

No sonic velocity data are available for the gaseous ClF_5 state.

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2.2.5 Electromagnetic Properties

The electrical, magnetic, and electromagnetic (optical) properties have been grouped as "Electromagnetic Properties." These properties generally are related to the electronic structure of the atoms in contrast to the transport properties which involve only molecular movement.

2.2.5.1 Index of Refraction. No data are available on the index of refraction of solid, liquid, or gaseous ClF_5 .

2.2.5.2 Dipole Moment. No data are available on the dipole moment of ClF_5 .

2.2.5.3 Dielectric Constant. The dielectric constant of liquid ClF_5 was measured from -80 to -17 C (Ref. 6). A curve-fit of the experimental data, shown in Fig. 10, can be expressed by the following equation:

$$\epsilon = -0.015 T_{(C)} + 3.08$$

The dielectric constant of gaseous ClF_5 , experimentally measured at ~10 megacycles, 23.9 C, and 746.5 mm Hg, was 1.00279 ± 0.00007 (Ref. 6).

2.2.5.4 Electrical Conductivity. The specific conductivity of liquid ClF_5 was found to be $<1.3 \times 10^{-9} \text{ ohm}^{-1} \text{ cm}^{-1}$ over a temperature range of -80 to -17 C (Ref. 6). The value at -17 C (1.4 F) was $0.45 \times 10^{-9} \text{ ohm}^{-1} \text{ cm}^{-1}$ (mho/cm).

2.2.5.5 Magnetic Susceptibility. No data are available on the magnetic susceptibility of ClF_5 .

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TABLE 4

PHYSICAL PROPERTIES OF ClF_5

Property	Value		Figure Number	Reference Number
	Metric	English		
<u>General Identification</u>				
Identification		CPF, Compound A, Fluoridyne		3
Molecular Formula		ClF_5		3
Molecular Weight		130.5 (calculated)		3
Freezing Point	-103 \pm 4 C	-153.4 \pm 2.2 F		3
Normal Boiling Point	-13.7 C	7.3 F		5
Critical Constants				
Temperature	143 \pm 0.5 C	289.4 \pm 0.9 F		6
Pressure	52.3 atm	771 psia		6
Density	0.565 gm/cc	35.5 lb/ft ³		6
Volume	1.77 cc/gm	0.0282 ft ³ /lb		6
<u>Phase Properties</u>				
Density				
Solid				
Liquid				
Gas			1, 1A	6
Vapor Pressure				
Surface Tension				
	1.793 gm/cc at 20 C	111.9 lb/ft ³ at 68 F		
	3.31 atm at 20 C	48.7 psia at 68 F	2, 2A	6
	15.55 dynes/cm at 20 C	0.1065 x 10 ² lb/ft at 68 F	3, 3A	6

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TABLE 4
(Continued)

Property	Value		Figure Number	Reference Number
	Metric	English		
Coefficient of Thermal Expansion				
Solid				
Liquid				
Gas				
Compressibility				
Adiabatic	$1.4995 \times 10^{-4} \text{ atm}^{-1} \text{ at } 20 \text{ C}$	$1.0203 \times 10^{-5} \text{ psi}^{-1} \text{ at } 68 \text{ F}$	4, 4A	5
Isothermal	$3.04 \times 10^{-4} \text{ atm}^{-1} \text{ at } 20 \text{ C}^*$	$2.07 \times 10^{-5} \text{ psi}^{-1} \text{ at } 68 \text{ F}^*$	5, 5A	9
Inert-Gas Solubility				
Nitrogen	$4.4 \times 10^{-4} \text{ gm/gm-atm at } 48.9 \text{ C}$	$3.0 \times 10^{-5} \text{ lb/lb-psi at } 120 \text{ F}$		10
Helium				
<u>Thermodynamic Properties</u>				
Heats of				
Formation	$-60.5 \text{ kcal/mole at } 25 \text{ C}$	$-834 \text{ Btu/lb at } 77 \text{ F}$		11
Fusion				
Vaporization	$5.513 \text{ kcal/mole at NBP}^*$	$76.04 \text{ Btu/lb at NBP}^*$		6
Heat Capacity				
Solid				
Liquid (C_s)	$38.78 \text{ cal/mole-K at } 20 \text{ C}$	$0.2972 \text{ Btu/lb-R at } 68 \text{ F}$	6, 6A	14

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TABLE 4
(Continued)

Property	Value		Figure Number	Reference Number
	Metric	English		
Gas				
Cp				
Cv				
Enthalpy				
Entropy				
<u>Transport Properties</u>				
Viscosity				
Liquid				
Gas	0.328 cp at 20 C	2.20×10^{-4} lb/ft-sec at 68 F	7, 7A	14
Thermal Conductivity				
Liquid				
Gas	0.4581×10^{-3} cal/cm-sec at 20 C*	0.1108 Btu/ft-hr-F at 68 F*	8, 8A	15
Velocity of Sound				
Liquid				
Gas	611.7 m/sec at 20 C	2007 ft/sec at 68 F	9, 9A	10
<u>Electromagnetic Properties</u>				
Index of Refraction				
Solid				
Liquid				
Gas				

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TABLE 4
(Concluded)

Property	Value		Figure Number	Reference Number
	Metric	English		
Dipole Moment				
Dielectric Constant				
Liquid	$\epsilon = 0.015 T_{(C)} + 3.08$ (from -80 to -17 C)			6
Gas	0.0279 at 23.9 C (75 F), 10 mc, 746.5 mm Hg		10	6
Electrical Conductivity	0.45×10^{-9} mho/cm at -17 C (1.4 F)			6
Magnetic Susceptibility				

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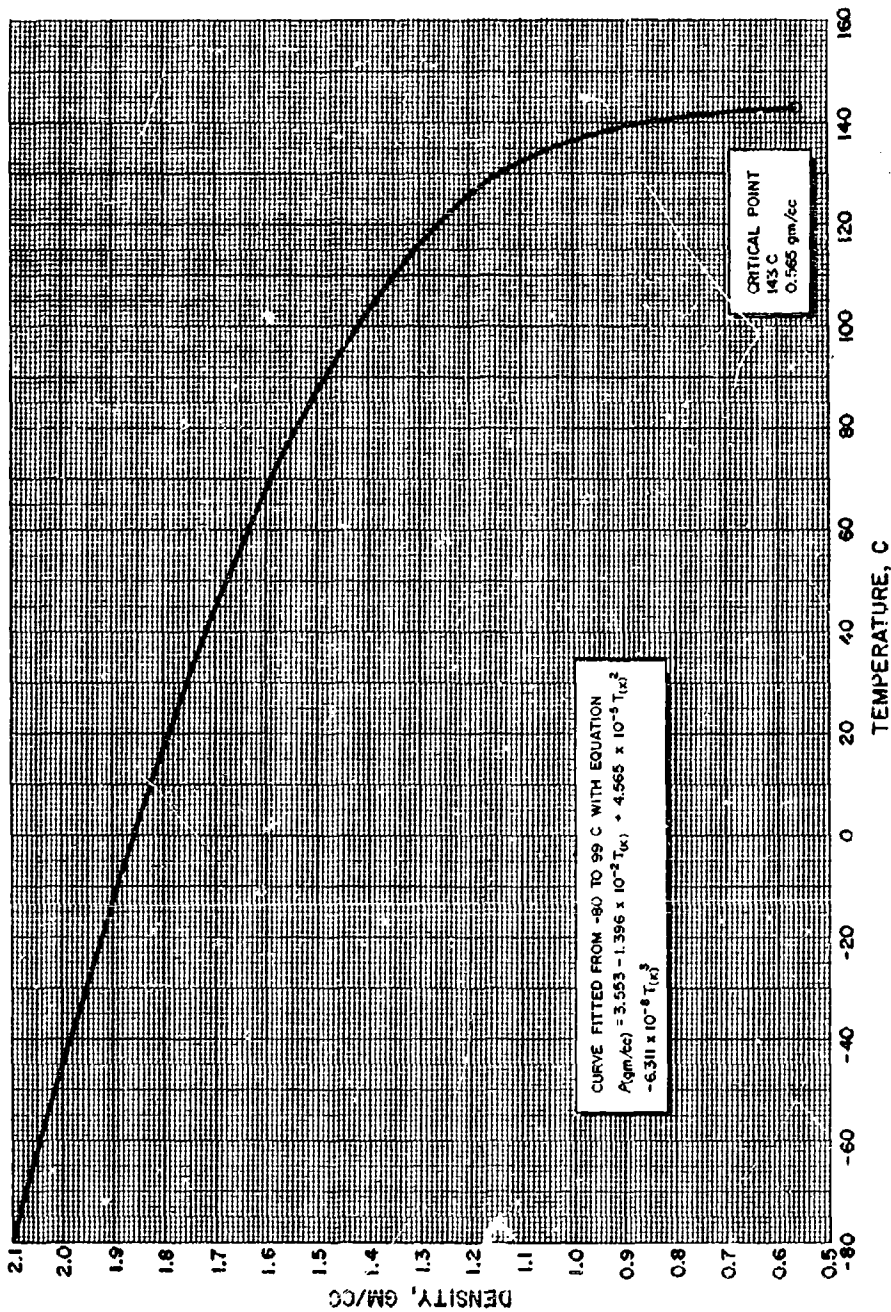


Figure 1. Density of Liquid Chlorine Pentafluoride (Ref. 6)

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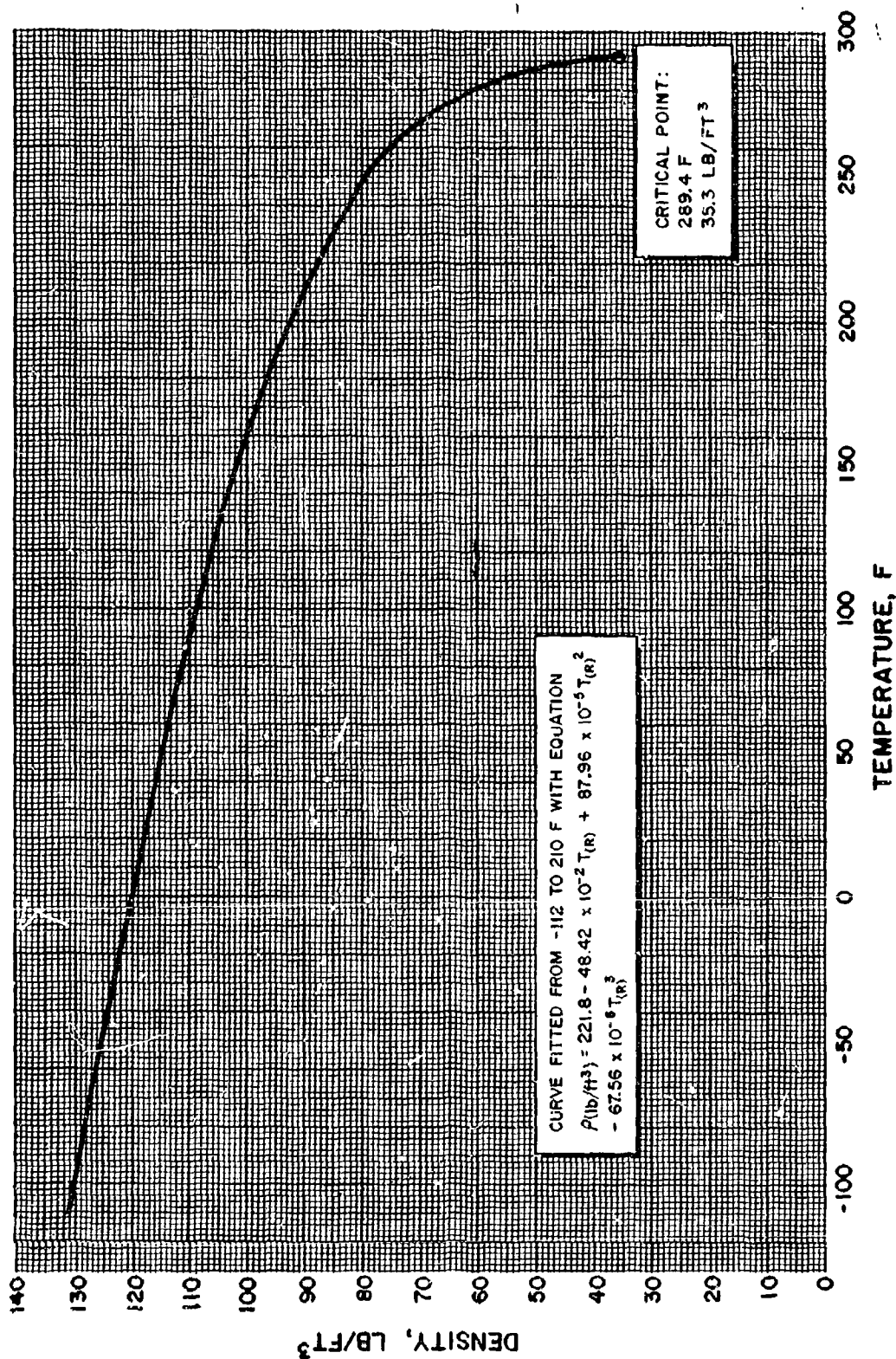


Figure 1A. Density of Liquid Chlorine Pentafluoride (Ref. 6)

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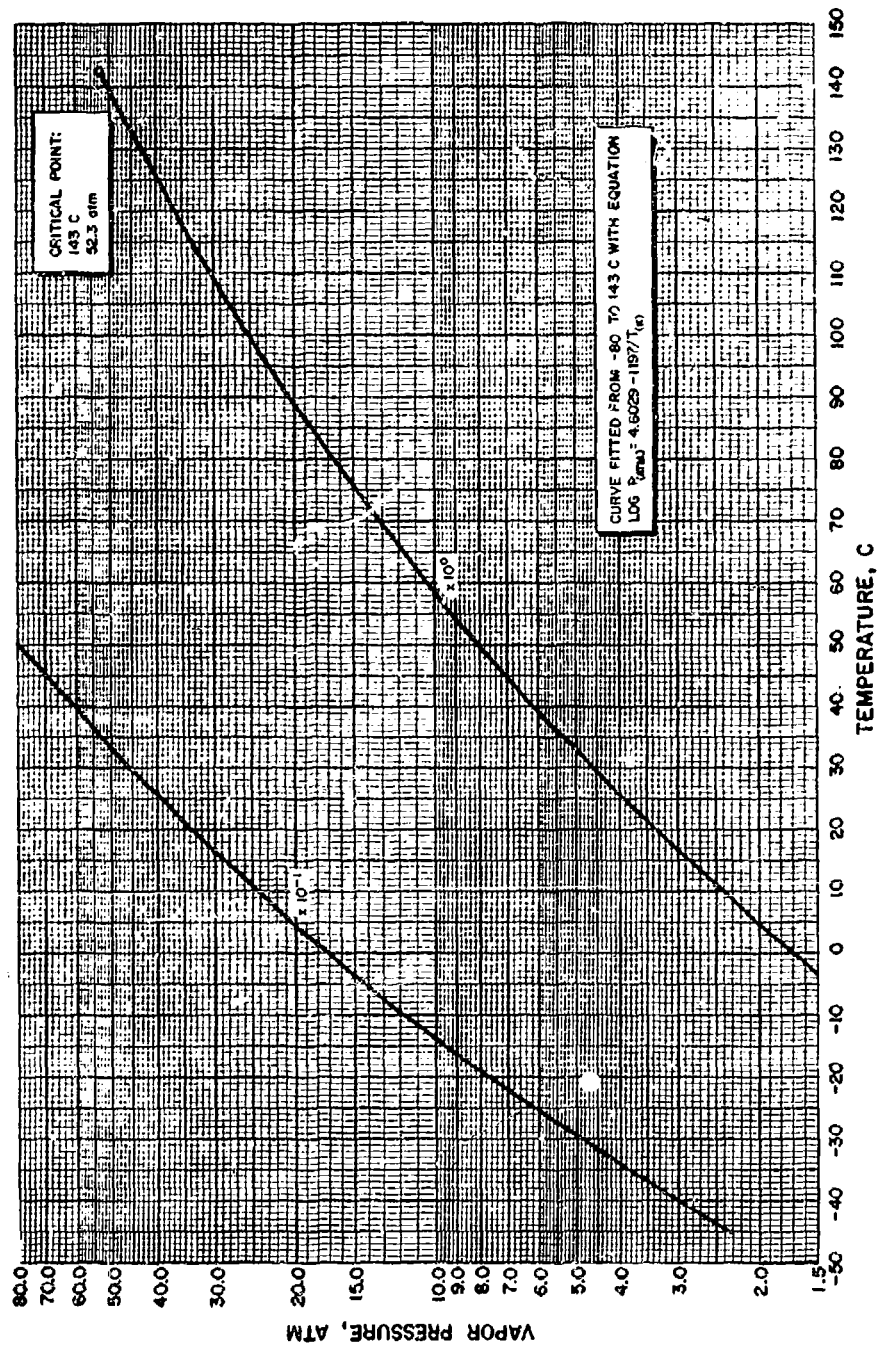


Figure 2. Vapor Pressure of Chlorine Pentafluoride (Ref. 6)

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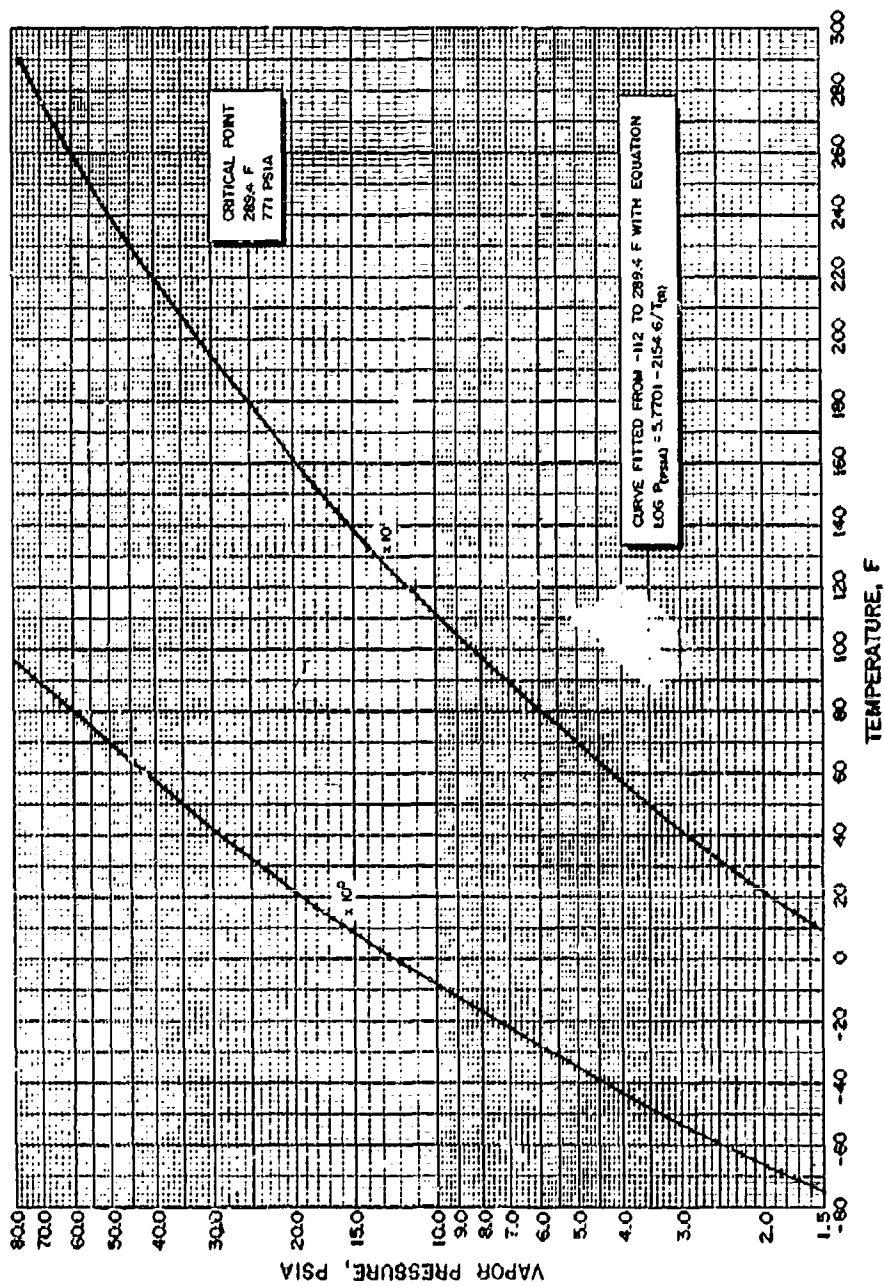


Figure 2A. Vapor Pressure of Chlorine Pentafluoride (Ref. 6)

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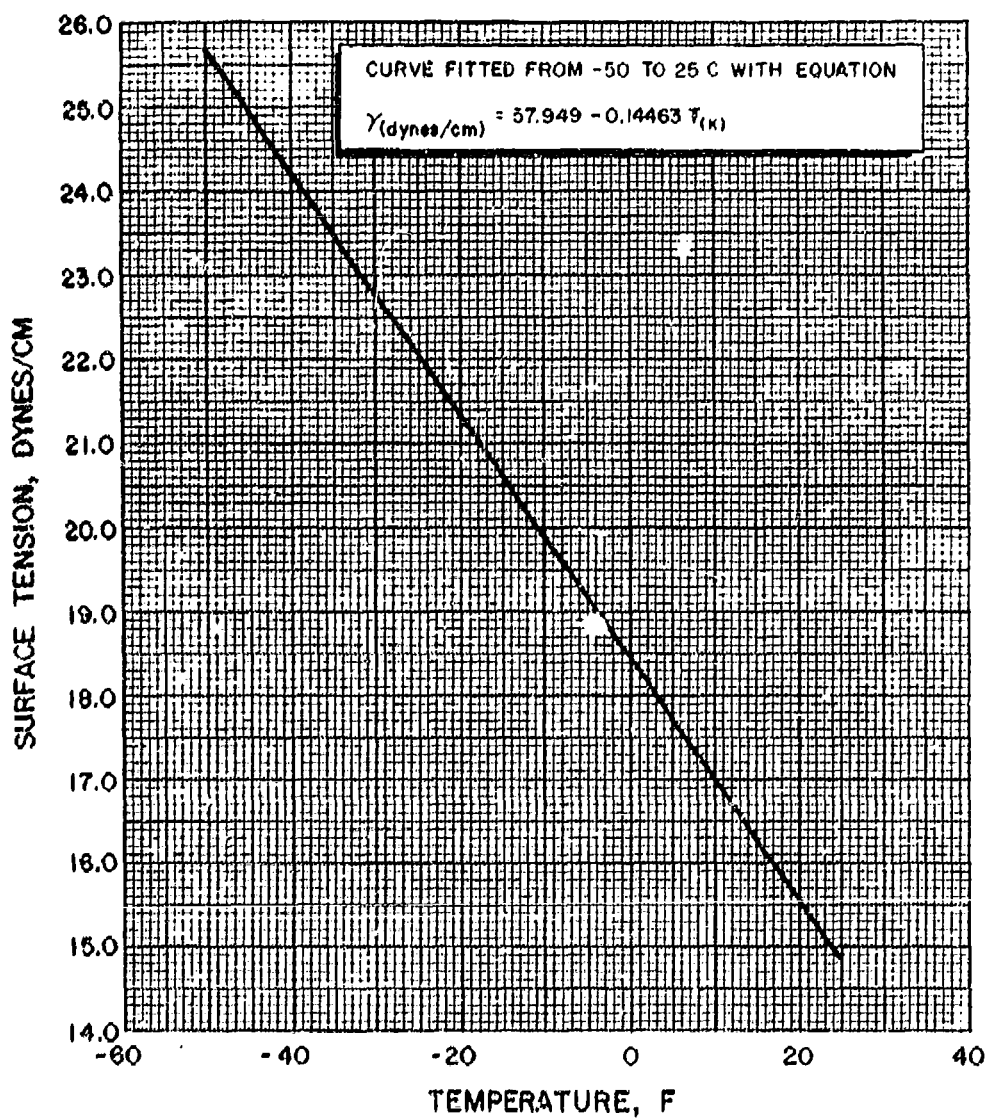


Figure 3. Surface Tension of Chlorine Pentafluoride (Ref. 6)

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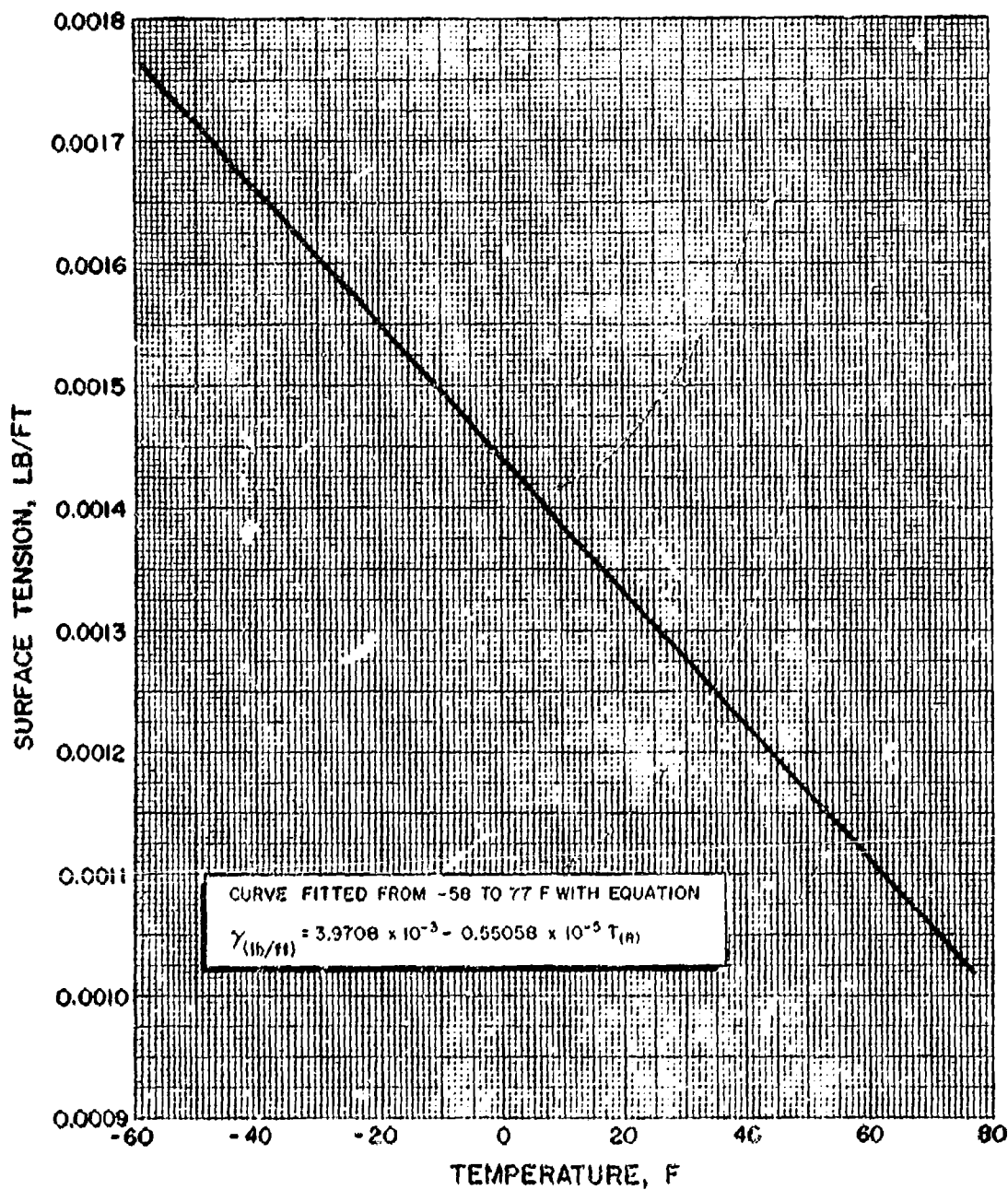


Figure 3A. Surface Tension of Chlorine Pentafluoride (Ref. 6)

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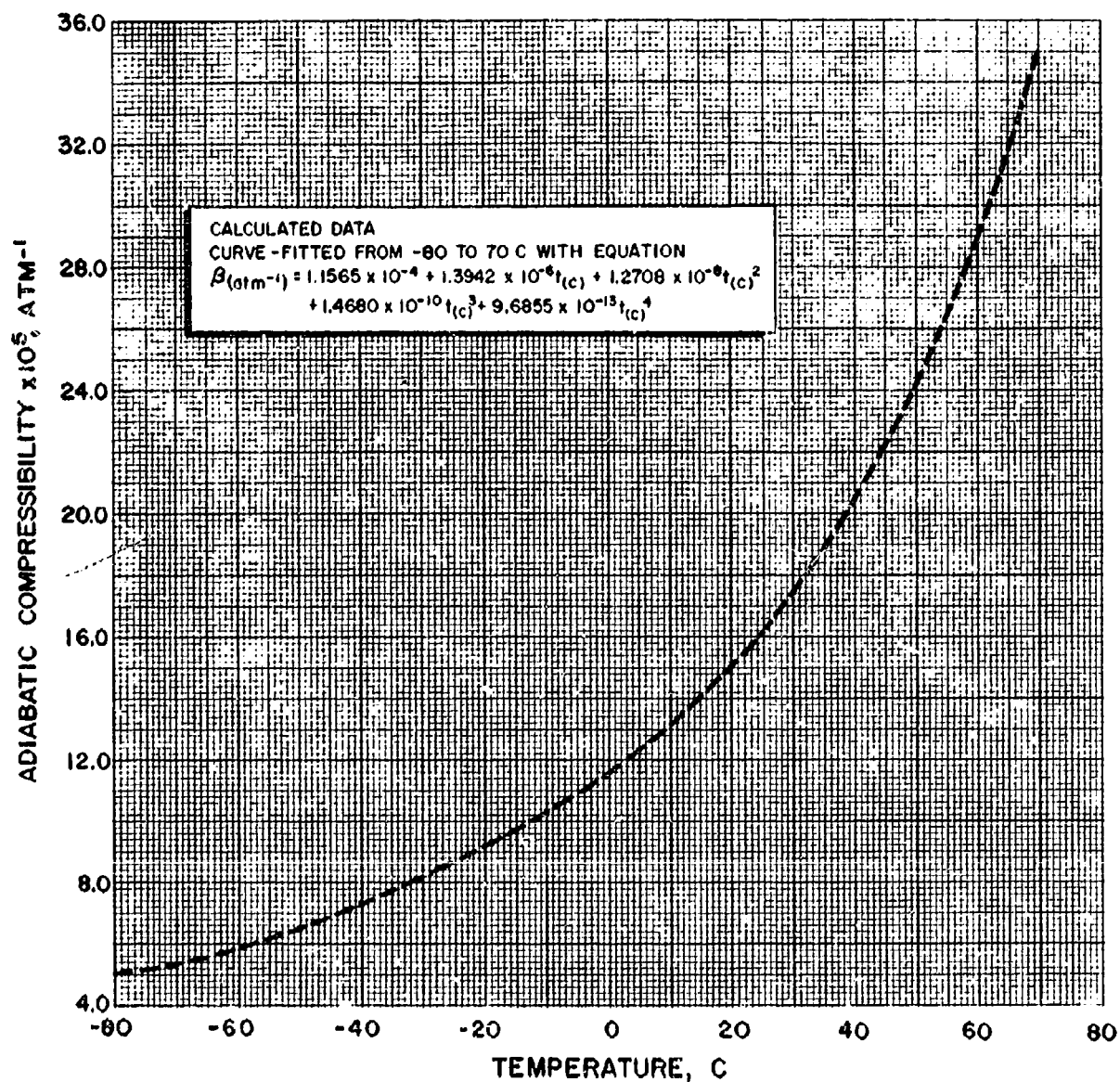


Figure 4. Adiabatic Compressibility of Chlorine Pentafluoride (Ref. 5)

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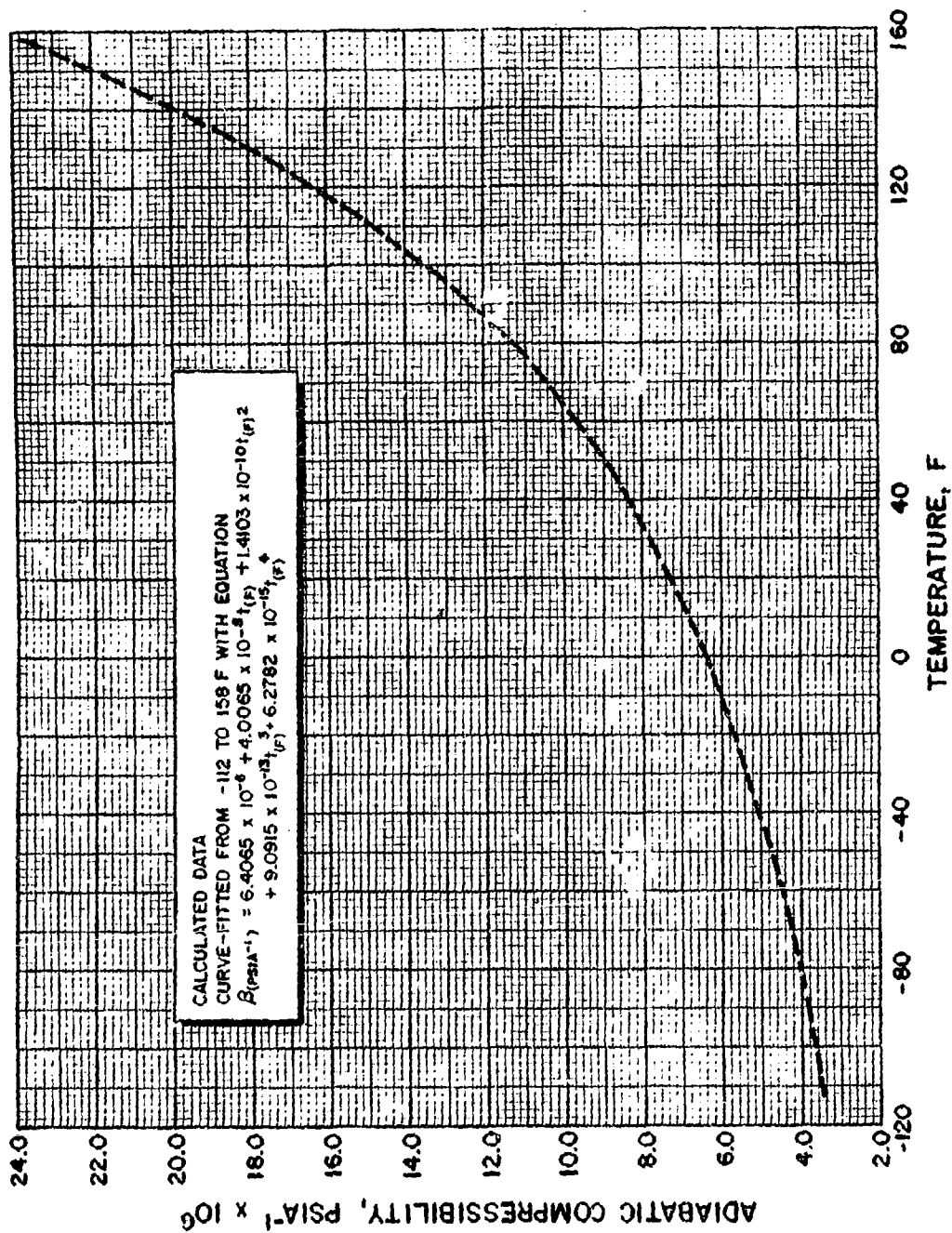


Figure 4A. Adiabatic Compressibility of Chlorine Pentafluoride (Ref. 5)

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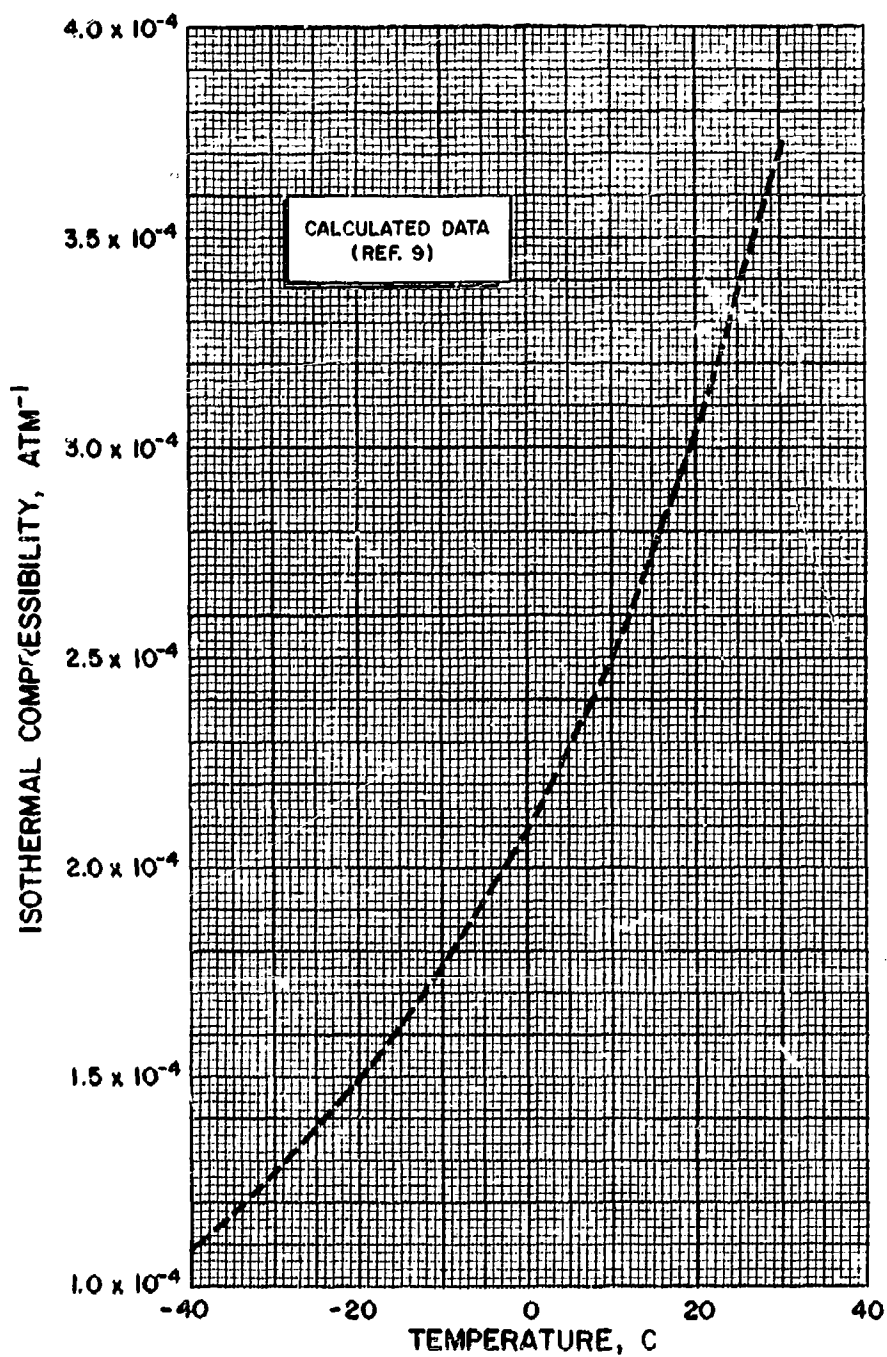


Figure 5. Isothermal Compressibility of Chlorine Pentafluoride

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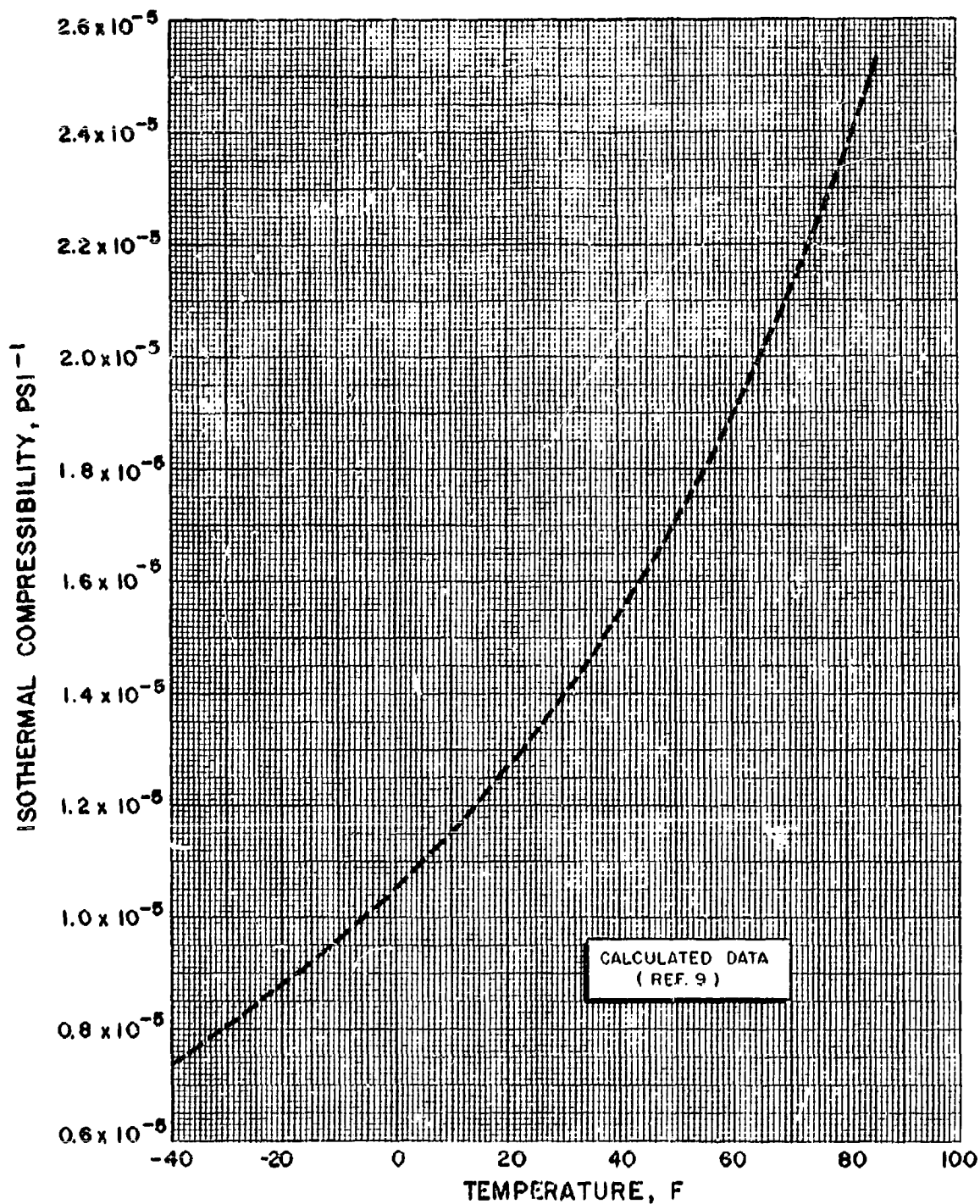


Figure 5A. Isothermal Compressibility of Chlorine Pentafluoride

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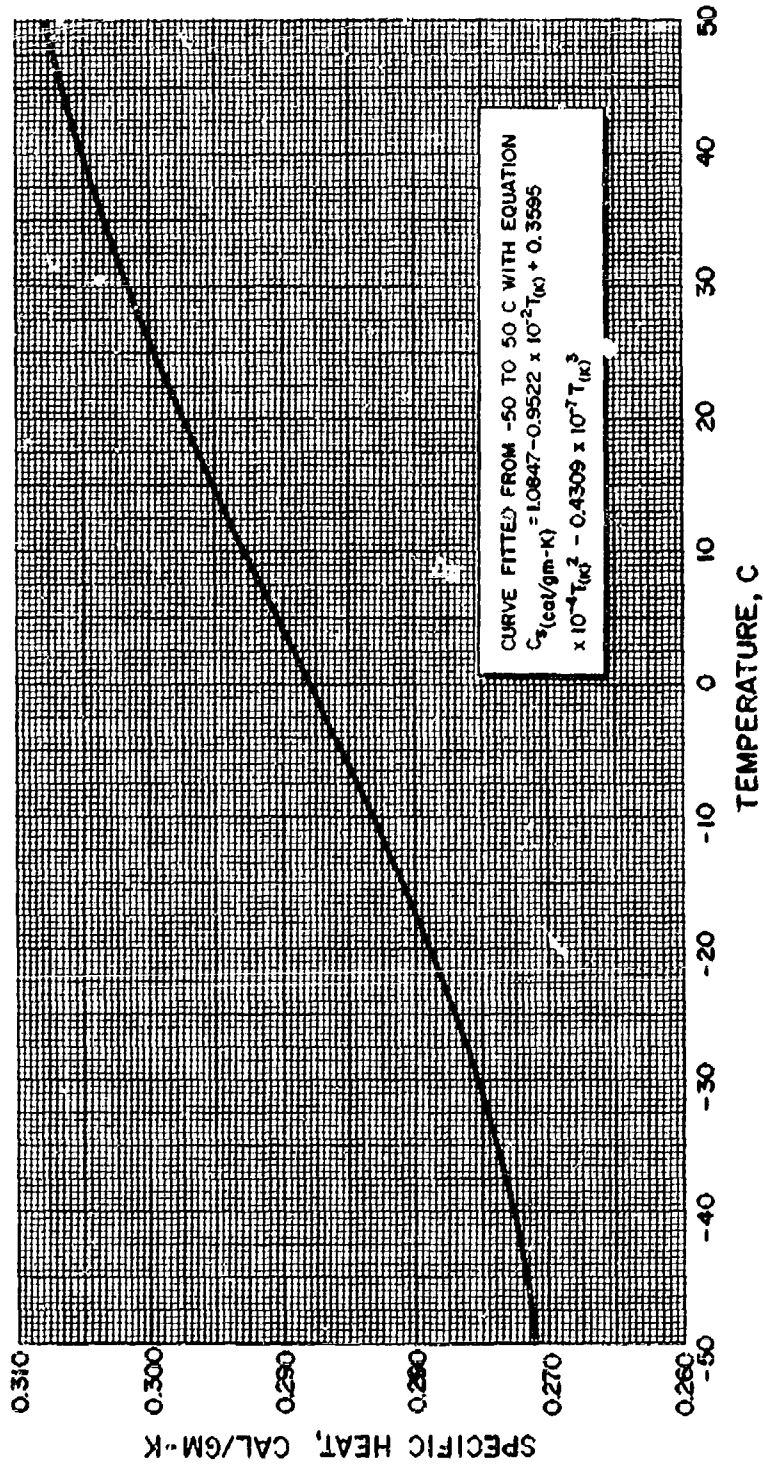


Figure 6. Specific Heat of Chlorine Pentafluoride (Ref. 14)

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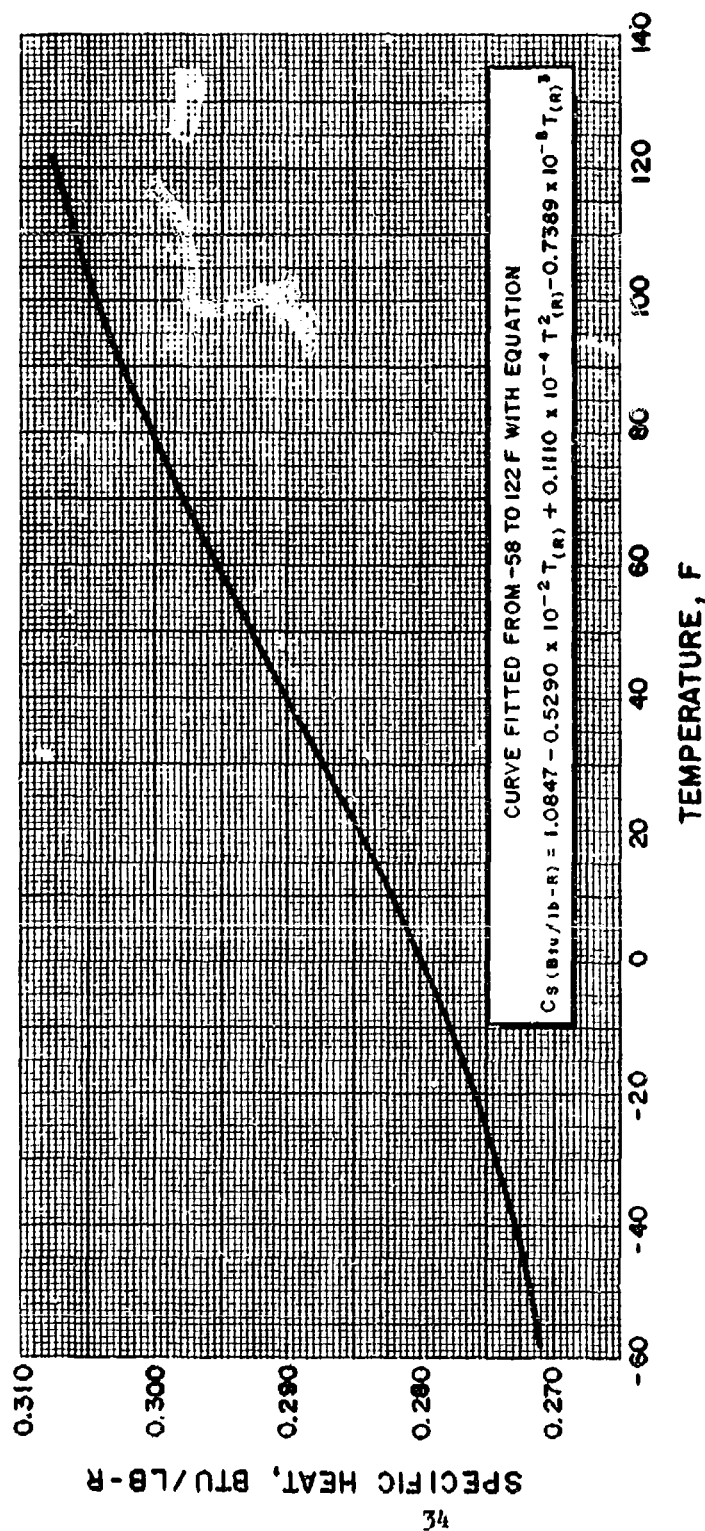


Figure 6A. Specific Heat of Chlorine Pentafluoride (Ref. 18)

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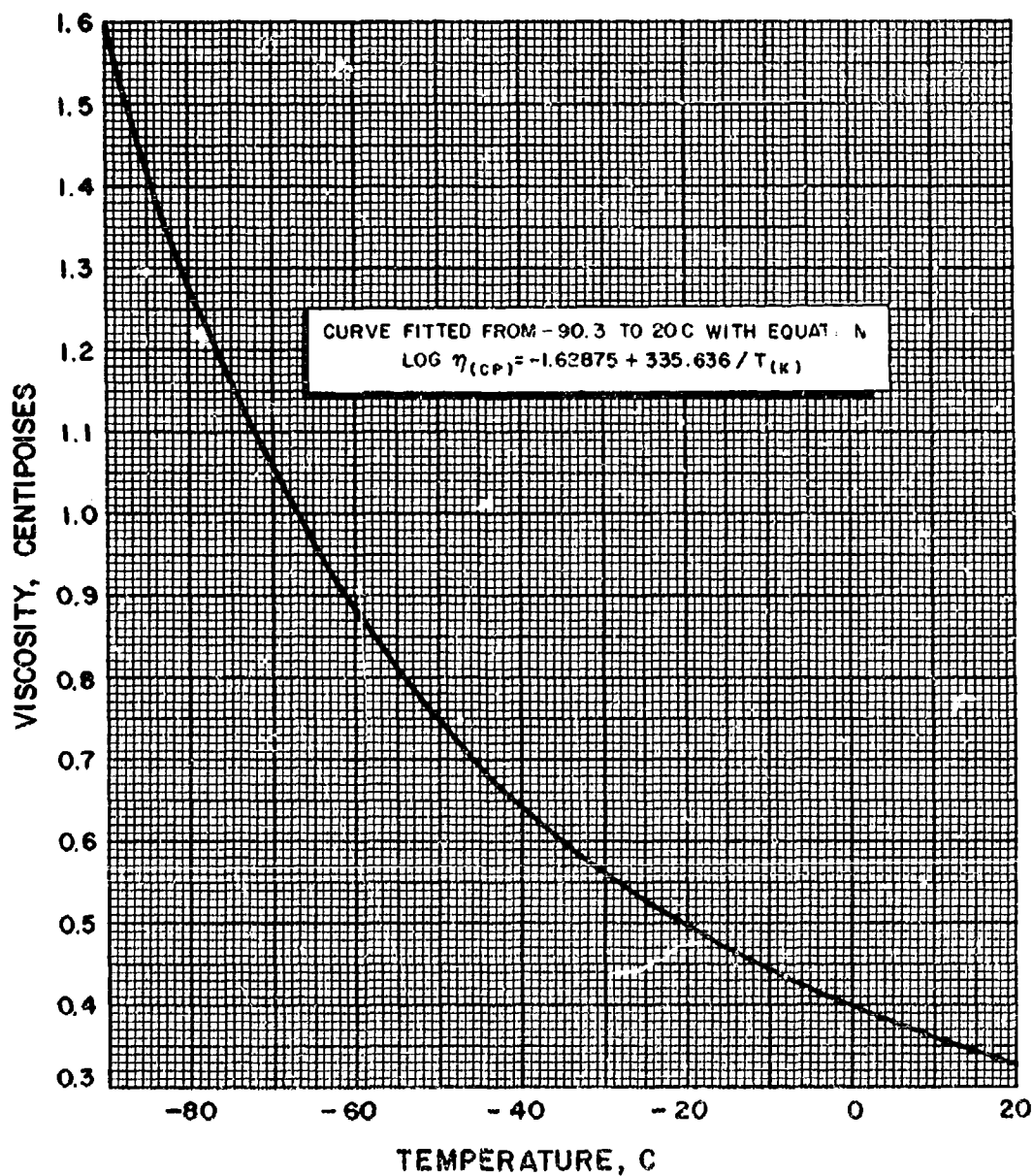


Figure 7. Viscosity of Chlorine Pentafluoride (Ref. 14)

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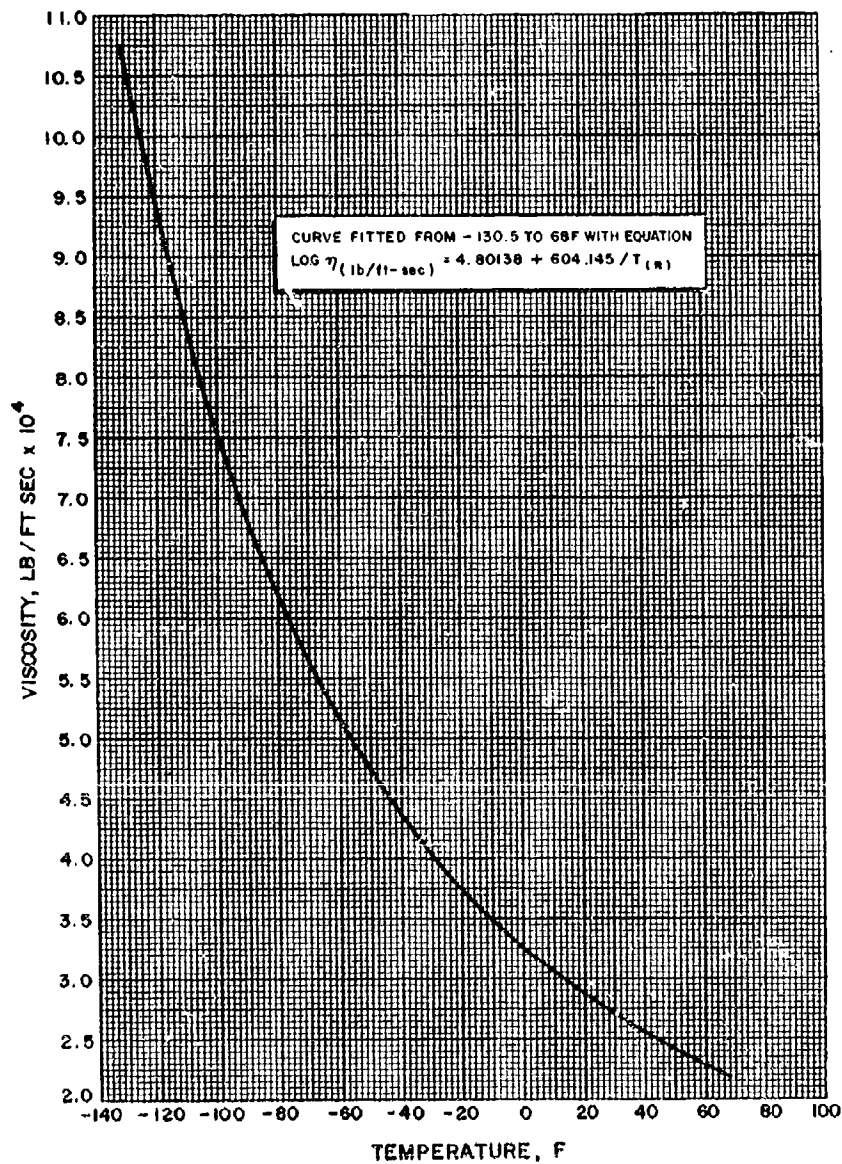


Figure 7A. Viscosity of Chlorine Pentafluoride (Ref. 14)

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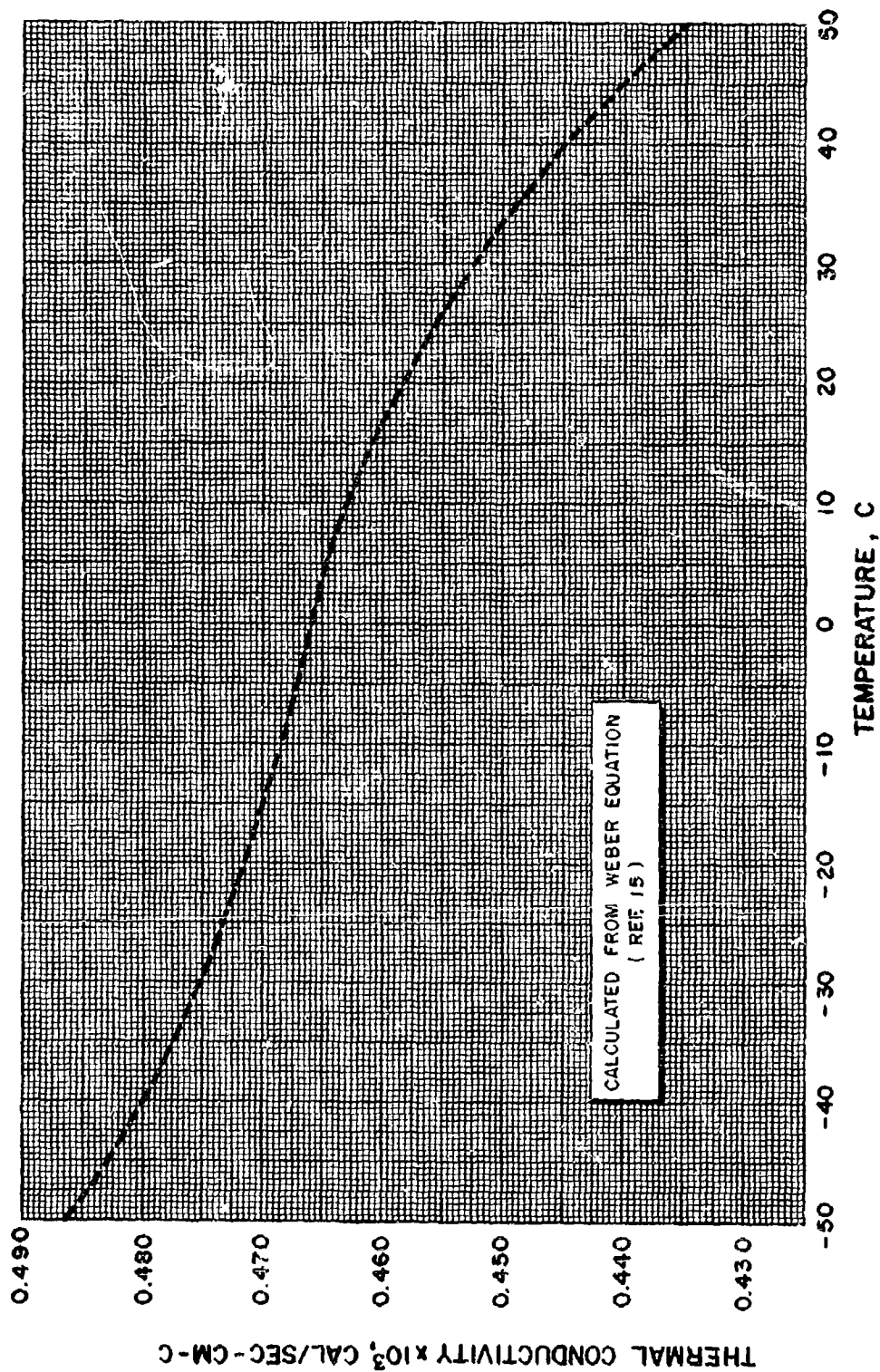


Figure 8. Thermal Conductivity of Chlorine Pentafluoride

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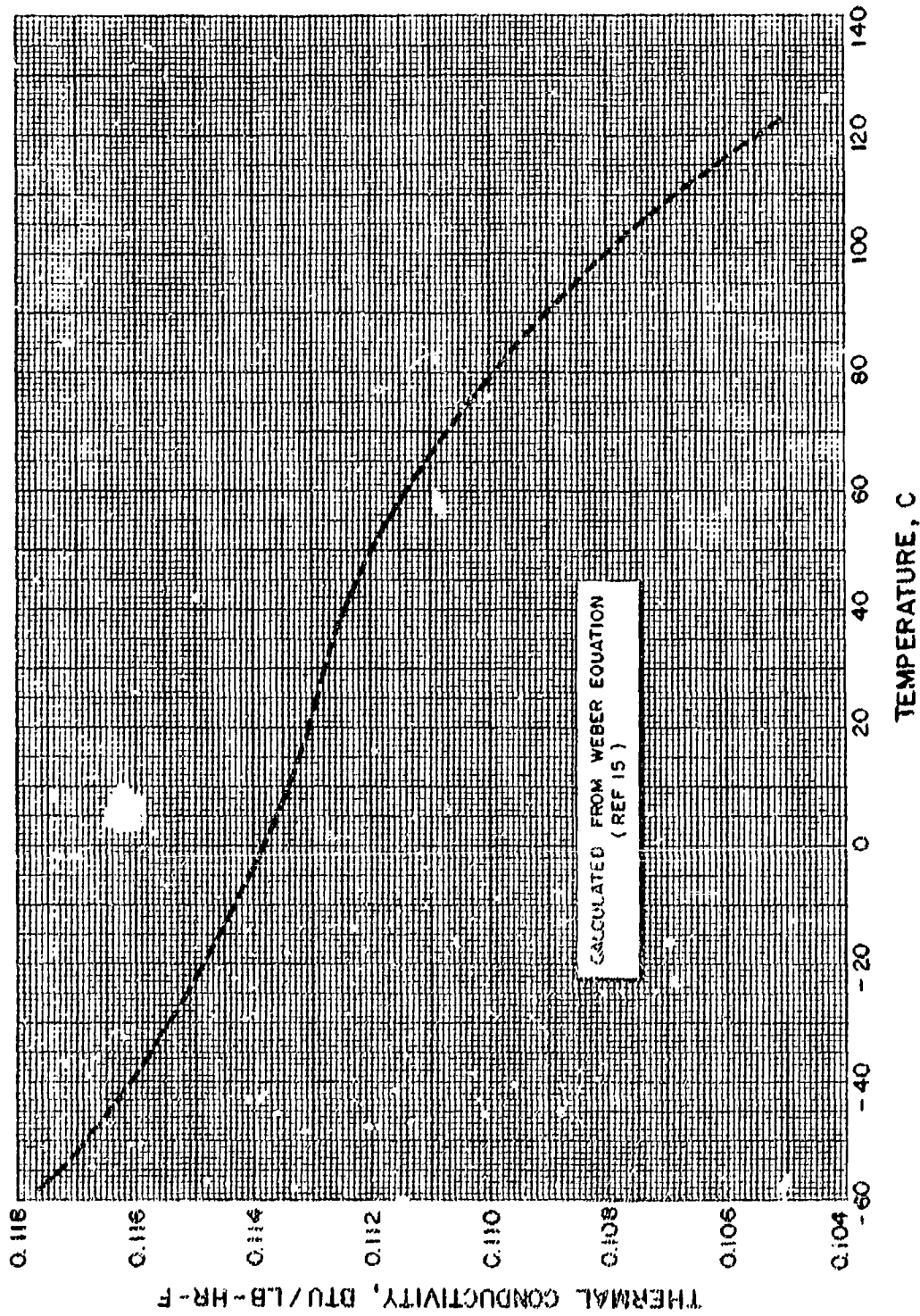


Figure 8A. Thermal Conductivity of Chlorine Pentafluoride

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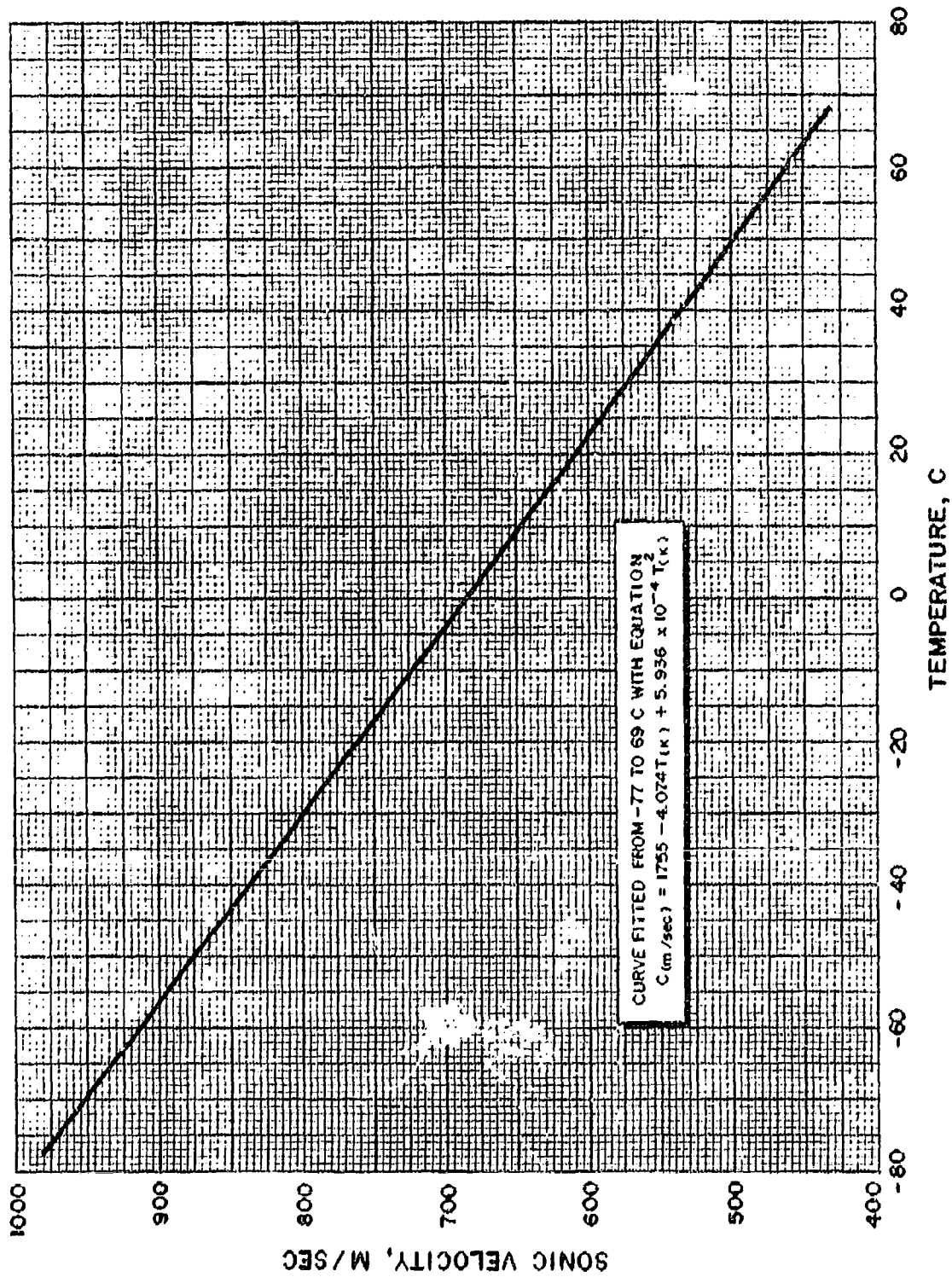


Figure 9. Sonic Velocity of Chlorine Pentafluoride (Ref. 10)

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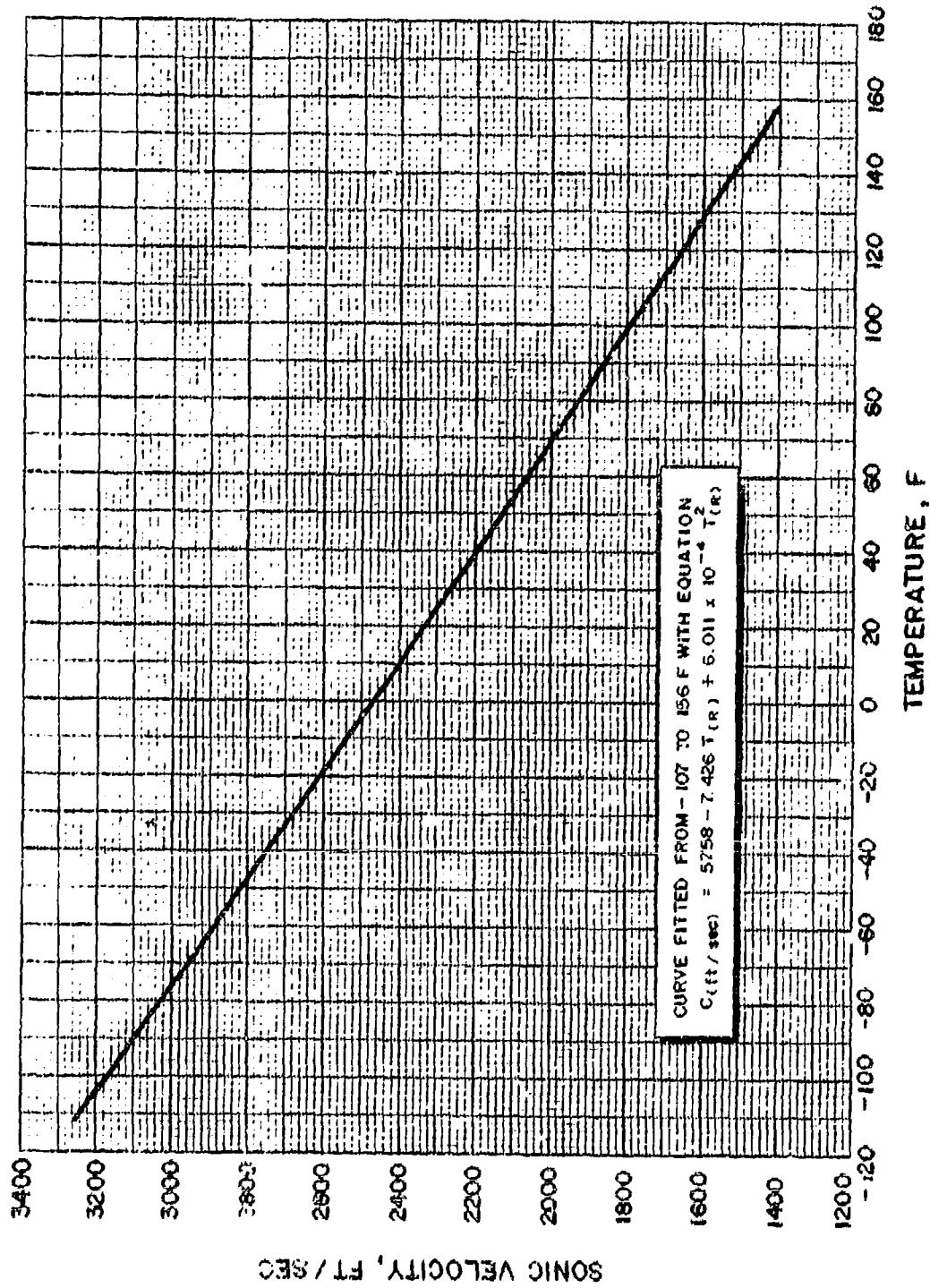


Figure 9A. Sonic Velocity of Chlorine Pentafluoride (Ref. 10)

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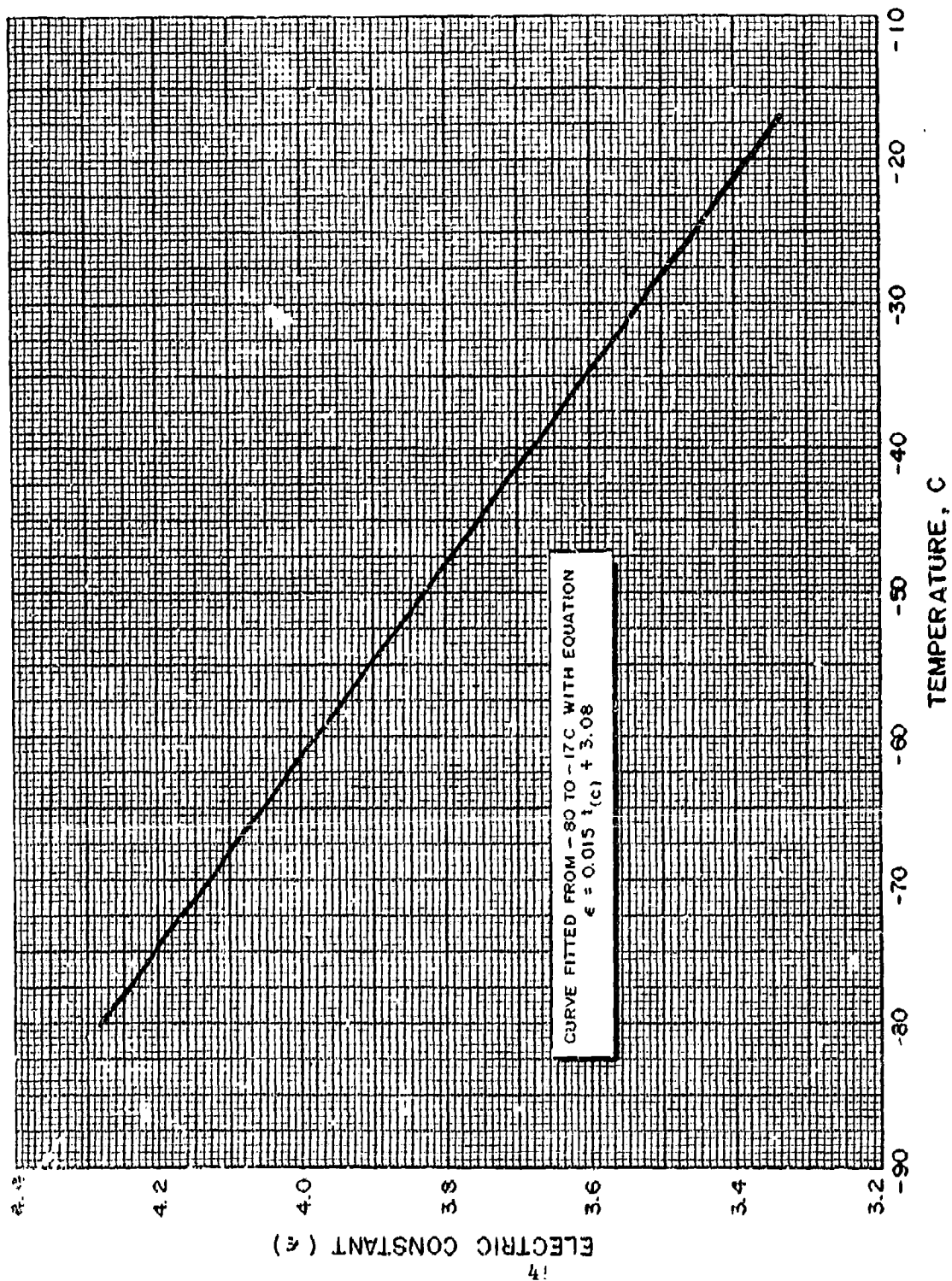


Figure 10. Dielectric Constant of Chlorine Pentafluoride (Ref. 6)

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2.3 CHEMICAL PROPERTIES

The chemical characterization of ClF_5 has been conducted by a number of investigators with various objectives. A primary effort (Ref. 8) was directed toward definition of the chemistry of ClF_5 in relation to its use as a propellant to provide a sound basis for future operational propellant handling procedures. Other investigators (Ref. 6 and 13) have contributed toward the definition of chemical properties through propellant characterization and engineering studies. A less extensive study (Ref. 16) has provided additional solubility and chemical reactivity data, while various other investigators (Ref. 17 through 32) have contributed to the chemistry of ClF_5 through attempted synthesis of new compounds using ClF_5 as a starting or immediate compound. The results of these studies are summarized by various classes of elements and compounds in Table 5. Although the more interesting features regarding ClF_5 chemical reactivity are presented in the following paragraphs, more detailed data from these studies can be located in the appropriate references.

2.3.1 Reaction with Nontransition Metals

Results of reaction studies between ClF_5 and the nontransition metals (Groups IA, IIA, and IIIA of the Periodic Table) indicate the apparent nonreaction of Al powder, Mg powder, and Be chunks at 150 C over a 16-hour period.

2.3.2 Reaction with Transition Metals

Reaction studies of ClF_5 with the transition metals indicate that some correlations can be established between the completeness or degree of reactivity of the transition metals and the volatility or melting point of the resulting fluoride. Transition metals that form volatile or low melting fluorides react completely at

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150 C (and many react at ambient temperature). For example, vanadium, niobium, molybdenum, tantalum, and tungsten, all of which form fluorides melting below 120 C, react completely at ambient temperature. Titanium reacts more slowly under similar conditions. Transition metals (Cr, Mn, Co, Ni, Pd, Pt, Cu, Zn, and Cd), which are least reactive with ClF_5 in studies to 150 C, all form fluorides with melting points above 850 C.

2.3.3 Reactions with Nonmetals and Metalloids

The reactions between nonmetals or metalloids and ClF_5 appear to be typical for the action of interhalogen fluorides in general toward these reagents. All physical states of graphite react at ambient temperature. The extreme reactivity of activated charcoal is attributed to the high concentration of organic material normally found in charcoals.

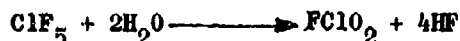
2.3.4 Reaction with Metal Oxides

For the most part, the reactivity of metal oxides with ClF_5 is similar to the reactivity of the parent metal. For example, the nontransition metal oxides (BeO , MgO , Al_2O_3 , PbO , and Bi_2O_3) are unreactive to 150 C. This relationship is also generally observed with transition metal oxides with two notable exceptions: HgO and CrO_3 . Both Cr_2O_3 and CrO_3 react completely at 150 C although chromium metal is unreactive under the same conditions. These data suggest that the formation of a stable, fluoride coating is not the only criterion for nonreactivity of a material with ClF_5 .

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2.3.5 Reaction with Water

Under carefully controlled conditions (and excluding metal), the reaction of ClF_5 with H_2O occurs according to the following equation:



With a local excess of water (in the presence of metal which provides a surface to catalyze the decomposition of FClO_2) the reaction apparently occurs as follows:

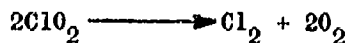


It is apparent from the second equation (which represents normal reaction conditions) that ClO_2 , an unstable and extremely sensitive species, is normally produced during the hydrolysis of ClF_5 .

Water in the form of metal fluoride hydrates reveals no difference in reactivity with respect to the products formed.

2.3.6 Reaction with Hydroxides

The reactivity of basic, neutral, and acidic hydroxides toward ClF_5 is similar in that HF and O_2 are formed in all cases. The formation of Cl_2 and/or ClO_2 from these reactions is a result of the degradation of ClF_5 , except in the cases of $\text{Na}_2\text{B}_4\text{O}_7 \cdot 10\text{H}_2\text{O}$ and $\text{Na}_2\text{SiO}_3 \cdot 9\text{H}_2\text{O}$. The initial reaction of hydroxides appears to be the formation of HF. Chlorine, generated from the decomposition of ClO_2 , accounts for the formation of $\text{Ca}(\text{OCl})_2$ in the reaction of ClF_5 and $\text{Ca}(\text{OH})_2$ as follows:



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Similar chlorination reactions also probably occur in other ClF_5 -hydroxide reactions.

2.3.7 Reaction with Solvents

Chlorine pentafluoride compatibility and solubility studies (Ref. 8) with hydrogen fluoride and halocarbon-base solvents indicate Freon 11, Freon 113 and Hooker Fluoride Oil are compatible and miscible with ClF_5 at ambient temperature. At -30°C , however, Freon 113 is not miscible. The tertiary amine, $(\text{C}_4\text{F}_9)_3\text{N}$, and the cyclic perfluoroethers, FC-75 and FC-77, are miscible with ClF_5 in 50 m/o mixtures at ambient temperature. At -30°C , no solubility is apparent with the tertiary amine. No F^{19} exchange is apparent in the halocarbon solutions at 27°C . Anhydrous HF will dissolve 6 m/o ClF_5 at -38°C and greater than 13 m/o at 0°C . No F^{19} exchange is observed in the HF- ClF_5 system which precludes any appreciable solvent-solvent interaction and particularly excludes the equilibrium:



Chloroform and carbon tetrachloride both react slowly with ClF_5 at ambient temperature to form Cl_2 and CFCl_3 . In addition to these products, the reaction with chloroform also forms CHCl_2F , CCl_2F_2 , and CF_4 . Because of this reaction, no ClF_5 solubility data are available with CCl_4 and CHCl_3 .

2.3.8 Miscellaneous Reactions

The chemical reactivity of ClF_5 with a number of reagents encompassing additional categories of compounds are also presented in Table 5. These include metalloid oxides, nonmetal oxides, salts of oxyacids, halides, borides, carbides, nitrides, sulfides,

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Lewis acids and bases, etc. Because the chemistry of these systems are of special interest to a small minority (for example, the interaction of ClF_5 with Lewis acids is only of academic interest), investigators interested in the descriptive aspects of these reactions, such as characterization and properties, should refer to the cited original sources.

From a practical point of view, engineers (or chemists) will not encounter the problem of "treating" these miscellaneous reagents with ClF_5 . Should a special situation develop, again the original literature should be consulted.

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TABLE 5

REACTIVITY OF ELEMENTS AND COMPOUNDS WITH ClF_5

Material	Test Temperature	Results and Products	Reference Number
<u>Metals and Metalloids</u>			
Li (chunk)	Ambient	Reaction: LiF , Cl_2	8
Be (chunk)	Ambient	No reaction	8
	150 C	No reaction	8
Mg (powder)	Ambient	No reaction	8
	150 C	No reaction	8
Ca (chunk)	Ambient	No reaction	8
	150 C	Partial reaction: flaking of CaF_2	8
B (granular)	Ambient	Reaction: BF_3 , Cl_2	8
Al (powder)	Ambient	No reaction	8
	150 C	No reaction	8
In (Powder)	Ambient	No reaction	8
	150 C	Extensive surface attack and film	8
Ge (wafer)	Ambient	Reaction: GeF_4	8
Sn (powder)	Ambient	No reaction	8
	150 C	Reaction: SnF_4 , Cl_2	8
Pb	Ambient	No reaction	8
	150 C	Surface attack	8
As (chunk)	Ambient	Reaction: AsF_5 , Cl_2	8
Sb (chunk)	Ambient	Reaction: SbF_5 , $\text{ClF}_4^+ \text{SbF}_6^-$	8
Bi (powder)	Ambient	No reaction	8
	150 C	Surface attack	8

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TABLE 5
(Continued)

Material	Test Temperature	Results and Products	Reference Number
<u>Transition Metals 3d Series</u>			
Cu (wire)	Ambient	No reaction	8
	150 C	No reaction	8
Zn (granular)	Ambient	No reaction	8
	150 C	No reaction	8
Ti (chunks)	Ambient	Slow reaction; complete reaction in 1 week; TiF_4 , Cl_2 , coating	8
	150 C	One-half reaction in 1 day	8
V (granular)	Ambient	Reaction: Cl_2	8
Cr (chunks)	Ambient	No reaction	8
	150 C	No reaction	8
Mn (chips)	Ambient	No reaction	8
	150 C	No reaction	8
Fe (powder)	105 C	No reaction	8
	150 C	Reaction: FeF_3 , Cl_2	8
Co (rod) (powder)	Ambient	No reaction	8
	150 C	No reaction	8
Ni (sheet)	Ambient	No reaction	8
	150 C	No reaction	8
<u>Transition Metals 4d, 5d Series</u>			
Ag (reagent-grade shot) (electrolytic plate)	Ambient	No reaction	8
	150 C	Severe surface attack: AgF , Cl_2	8

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TABLE 5
(Continued)

Material	Test Temperature	Results and Products	Reference Number
<u>Transition Metals 4d, 5d Series</u>			
Cd (rod)	Ambient	No reaction	8
Zr (chunks)	Ambient	No reaction	8
	150 C	Surface darkened	8
Nb (powder)	Ambient	Reaction: Cl_2	8
Mo (strips)	Ambient	Reaction: MoF_6 , Cl_2	8
Pd (sheet)	Ambient	No reaction	8
	150 C	Slight tarnish	8
Au (sheet)	Ambient	No reaction	8
	150 C	Surface tarnished	8
Hg	Ambient	Reaction: HgF_2 , HgCl_2	8
Ta (strips)	Ambient	Slow reaction: ClF_3 , TaF_5	8
	150 C	Reaction: purple coating	8
W (powder)	Ambient	Reaction: WF_6 , Cl_2	8
Pt (wire)	Ambient	No reaction	8
	150 C	No reaction	8
<u>Nonmetals</u>			
C (Lampblack Raven-30 1-Percent Volatiles)	Ambient	No reaction	8
	150 C	Partial reaction: Cl_2 , CF_4 , CF_3Cl	8
Si (chunks)	Ambient	Reaction: SiF_4 , Cl_2	8
Graphite (powder)	Ambient	Partial Reaction: absorption into lattice; no CF_4 , Cl_2	8

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TABLE 5
(Continued)

Material	Test Temperature	Results and Products	Reference Number
<u>Nonmetals</u>			
Graphite (pressed rod)	Ambient	10-percent uptake: intercalation compound formed; no CF_4 , Cl_2	8
Charcoal (activated)	-150 C	Extremely vigorous reaction: CF_4 , C_2F_6 , SF_6	8
Lamblack (Neo-Supra II; 15-percent volatiles)	Ambient	Reaction: CF_4 , CF_3Cl , ClO_2 , C_2F_6	8
P_4 violet powder	< -80 C	Reaction: PF_5 , Cl_2	8
S_8 (roll excess)	Ambient	Reaction: SF_4 , Cl_2	8
Se_8 (powder, excess)	Ambient	Reaction: SeF_6 , Cl_2	8
N_2	-196 C	Solubility of solid ClF_5 < 0.1 percent	.
	25 C	No reaction	8
O_2 (dry)	-183 C	Solubility of solid ClF_5 < 0.1 percent	16
	Ambient	No reaction	8
	150 C	No reaction	8
	230 C	No reaction	8
	375 to 430 C	No reaction	26
O_3	-133 to 78 C	No reaction; some decomposition of O_3	16
F_2	-196 C	Solubility of solid ClF_5 < 0.1 percent	16
	310 C	No reaction	8
	300 to 375 C	No reaction	26

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TABLE 5
(Continued)

Material	Test Temperature	Results and Products	Reference Number
<u>Nonmetals</u>			
Cl_2	Ambient	No reaction	8
	150 C	No reaction	8
	>180 C	Partial reaction: $\text{ClF}_3 + \text{F}_2$	8
Br_2 (l)	100 C	Partial reaction: $\text{BrF}_3 + \text{Cl}_2$	8
I_2 (s)	Ambient	Reaction: IF_5 , ClF , ClF_3	8
ClF	-88 C	No reaction: homogeneous mixture	16
ClF_3	-83 C	Homogeneous mixture	16
ClO_2F	230 to 430 C	No reaction	26
ClO_3F	300 C	No reaction	28
NF_3	200 C	No reaction	31
N_2F_4	Ambient	No reaction	18
KrF_4	-93 to -53 C	No reaction	16
NOF	-78 C	Reaction: solid (complex)	17, 30
NO_2F	-78 C	No reaction	28
	-183 C	Solubility of solid $\text{ClF}_5 < 0.1$ percent	16
OF_2	-78 C	No reaction	17
	Ambient	No reaction	18
	300 C	No reaction	31
O_2F_2	-93 C	No reaction: some decomposition of O_2F_2	16

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TABLE 5
(Continued)

Material	Test Temperature	Results and Products	Reference Number
<u>Nonmetals</u>			
H ₂ O ₂ (90 percent)	-78 C	Violent reaction: ClO ₃ F, ClO ₂ , O ₂	17
(98 percent)	-78 C	Violent reaction: ClO ₃ F, ClO ₂ , O ₂	17
<u>Metal and Metalloid Oxides</u>			
Na ₂ O ₂ (powder)	Ambient	No reaction	8
	100 C	Reaction: ClF ₃ , FClO ₂ , O ₂	8
KO ₂ (powder)	Ambient	Reaction: ClF ₃ , FClO ₂ , ClO ₂ , O ₂	8
BeO (powder)	Ambient	Slight reaction	8
	150 C	Slight reaction: FClO ₃	8
MgO (powder)	Ambient	No reaction	8
	150 C	No reaction	8
CaO (powder)	Ambient	Complete reaction: O ₂ , Cl ₂	8
CaO ₂ (powder)	Ambient	Complete reaction: O ₂ , ClF ₃	8
BaO (powder)	Ambient	Reaction: FClO ₃ , O ₂ , Cl ₂ , ClO ₂	8
BaO ₂ (powder)	Ambient	No reaction	8
	100 C	Reaction: O ₂ , Cl ₂ , ClO ₂	8
B ₂ O ₃ (powder)	Ambient	Reaction: BF ₃ , O ₂ , Cl ₂ , FClO ₃ , BF ₄ ⁻	8
Al ₂ O ₃ (activated)	Ambient	Reaction: FClO ₃ , Cl ₂	8
Al ₂ O ₃ (vacuum baked)	Ambient	No reaction	8
	150 C	No reaction	8

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TABLE 5
(Continued)

Material	Test Temperature	Results and Products	Reference Number
<u>Metal and Metalloid Oxides</u>			
Al ₂ O ₃ (fused; Lucalux)	Ambient	No reaction	8
	150 C	No reaction	8
SnO (powder)	Ambient	No reaction	8
	150 C	Reaction	8
SnO ₂ (powder)	Ambient	No reaction	8
	150 C	Slight reaction	8
PbO (powder)	Ambient	No reaction	8
	150 C	Slight reaction	8
PbO ₃ (powder)	Ambient	No reaction	8
	150 C	Complete reaction	8
Pb ₃ O ₄	Ambient	Complete reaction: Cl ₂ , O ₂	8
As ₂ O ₃ (powder)	Ambient	Complete reaction: AsOF ₃ , Cl ₂ , O ₂	8
As ₂ O ₅ (powder)	Ambient	Complete reaction: Cl ₂ , O ₂ , nonoxidized solids ²	8
Sb ₂ O ₃ (powder)	Ambient	Complete reaction: O ₂ , solids	8
Sb ₂ O ₅ (powder)	Ambient	Complete reaction: O ₂ , solids	8
Bi ₂ O ₃ (powder)	Ambient	No reaction	8
	150 C	Slight reaction	8
<u>Transition Metal Oxides</u>			
Cu ₂ O	Ambient	No reaction	8
	150 C	No reaction	8

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TABLE 5
(Continued)

Material	Test Temperature	Results and Products	Reference Number
<u>Transition Metal Oxides</u>			
CuO	Ambient	No reaction	8
	150 C	No reaction	8
Ag ₂ O	Ambient	Reaction: FC10 ₂ , ClO ₂ , O ₂ , Cl ₂ , AgF	8
ZnO	Ambient	No reaction	8
	150 C	Slight reaction	8
CdO	Ambient	No reaction	8
	150 C	Slight reaction	8
HgO	Ambient	No reaction	8
	Ambient	Reaction: ClO ₂ , ClO ₂ F, ClO ₂ F ₃	18
	150 C	Incomplete reaction: FC10 ₂ , ClF ₃ , ClO ₂	8
TiO ₂	Ambient	No reaction	8
	150 C	Slight reaction: O ₂ , Cl ₂ , FC10 ₂ , TiF ₄	8
V ₂ O ₅	Ambient	No reaction	8
	150 C	Complete reaction: O ₂ , Cl ₂ , ClO ₂	8
Cr ₂ O ₃	Ambient	No reaction	8
	150 C	Extensive reaction: ClO ₂ , FC10 ₂	8
CrO ₃	Ambient	Complete reaction: CrO ₂ F ₂ , FC10 ₃ , Cl ₂ , O ₂	8
MoO ₃	Ambient	Complete reaction: FC10 ₃ , Cl ₂ , O ₂ , FC10 ₂ , ClO ₂	8
MnO ₂ (powder)	Ambient	No reaction	8
	150 C	No reaction	8

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TABLE 5
(Continued)

Material	Test Temperature	Results and Products	Reference Number
<u>Transition Metal Oxides</u>			
FeO	Ambient	No reaction	8
	150 C	Reaction: Cl_2 , FeClO_3 , O_2	8
Fe_2O_3	Ambient	No reaction	8
	150 C	Reaction: Cl_2 , O_2 , FeClO_3	8
Co_2O_3	Ambient	No reaction	8
	150 C	No reaction	8
NiO	Ambient	No reaction	8
	150 C	No reaction	8
Ni_2O_3	Ambient	No reaction	8
	150 C	No reaction	8
<u>Nonmetal Oxides</u>			
CO	Ambient	Complete reaction: COF_2 , COClF	8
CO_2	Ambient	No reaction	8, 18
SiO_2	-20 C and ambient	ClF_3 , Cl_2 , SiF_4 , SiO_2 , O_2 , ClO_2 , Cl_2O , ClOF	16
(quartz) (325-mesh)	Ambient	No reaction	8
	Ambient	No reaction	8
	Ambient	Reaction	18
	140 C	No reaction	31
	250 C	Reaction: SiF_4 , ClF_3 , O_2 , ClO_2F	31
NO	Ambient	Complete reaction: $\text{FNO} + \text{Cl}_2$	8

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TABLE 5
(Continued)

Material	Test Temperature	Results and Products	Reference Number
<u>Nonmetal Oxides</u>			
NO ₂	Ambient	Complete reaction: FC10 ₂ , C10 ₂ , FNO ₂ , FNO	8
	Ambient	Reaction: FNO, FNO ₂ , FNO ₃ , ClF ₃	18
P ₂ O ₅	Ambient	Complete reaction: POF ₃ , O ₂ , Cl ₂	8
SO ₂	Ambient	Vigorous reaction: SF ₆ , SOF ₄ , SOF ₂ , SO ₂ F ₂ , C10 ₂ , C10 ₃ F	18
SO ₃	Ambient	Complete reaction: Cl ₂ , SO ₂ F ₂ , O ₂	8
	Ambient	Explosive reaction	18
SOF ₄	Ambient	Slight reaction: SOF ₂ , SO ₂ F ₂	18
S ₂ O ₆ F ₂	<130 C	No reaction	18
	130 C	Slight reaction	18
ClO ₂	Ambient	No reaction	8
	100 to 110 C	No reaction	32
I ₂ O ₅	-100 to -78 C	Reaction: IF ₅ , IF ₇ O ₂ , C10 ₃ F, C10 ₂	17
<u>Water and Metal Fluoride Hydrates</u>			
H ₂ O (vapor in N ₂ with metal/Kel-F system)	0 C	Cl ₂ , C10 ₂ , FC10 ₃	8

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TABLE 5
(Continued)

Material	Test Temperature	Results and Products	Reference Number
<u>Water and Metal Fluoride Hydrates</u>			
$\text{KF} \cdot 2\text{H}_2\text{O}$	Ambient	ClO_2 , FClO_2 , FClO_3	8
$\text{MgF}_2 \cdot x\text{H}_2\text{O}$	Ambient (partial reaction)	ClO_2 , FClO_2 , FClO_3	8
H_2O (50-percent solution in HF with all Kel-F system)	-30 C	FClO_2	8
$\text{CrF}_3 \cdot 3 \frac{1}{2} \text{H}_2\text{O}$	Ambient	ClO_2 , FClO_3 , Cl_2	8
$\text{CuF}_2 \cdot 2\text{H}_2\text{O}$	Ambient	ClO_2 , FClO_2 , FClO_3	8
H_2O (liquid)	Ambient	Vigorous reaction	13
H_2O (vapor)	Ambient	Vigorous reaction: ClO_2F , ClO_3F , HF	13
H_2O	Ambient	Violent reaction	18
H_2O	0 C	HF, Cl_2 , HClO_3 , O_2	16
<u>Hydroxides</u>			
$\text{Ca}(\text{OH})_2$ - $\text{ClF}_5(l)$	Ambient	O_2 , Cl_2 , FClO_2 , CaF_2 , $\text{Ca}(\text{ClO})_2$	8
$\text{Ca}(\text{OH})_2$ - $\text{ClF}_3(g)$	Ambient	O_2 , Cl_2 , FClO_2 , FClO_3 , CaF_2	8
$\text{B}(\text{OH})_3$	Ambient	O_2 , Cl_2 , HF_3	8
$\text{NaB}_4\text{O}_7 \cdot 10\text{H}_2\text{O}$	Ambient	O_2 , HBF_3OH , NaBF_4 , $\text{HCl}(\text{hydrated})$	8

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TABLE 5
(Continued)

Material	Test Temperature	Results and Products	Reference Number
<u>Hydroxides</u>			
$\text{Al}(\text{OH})_3$	Ambient	O_2 , ClO_2 , FClO_2 , AlF_3	8
$\text{Na}^+\text{CO}_2(\text{OH})^-$	Ambient	NaF , O_2 , CF_4 , CF_3Cl , Cl_2	8
$(\text{CF}_3)_2\text{C}(\text{OH})_2$	Ambient	O_2 , CF_2O , CF_4 , CF_3Cl , ClO_2	8
$\text{CF}_3\text{CO}(\text{OH})$	-23 C	O_2 , FClO_2 , CF_3CFO	8
	25 C	O_2 , FClO_2 , CF_3CFO , $(\text{CF}_3\text{CO})_2\text{O}$	8
$\text{Na}_2\text{SiO}_3 \cdot 9\text{H}_2\text{O}$	Ambient	O_2 , Na_2SiF_6 , HCl (hydrated)	8
$\text{NO}_2(\text{OH})$ (70 percent)	Ambient	O_2 , Cl_2 , ClO_2 , FClO_2 , NO_2	8
$\text{K}^+\text{PO}_2(\text{OH})_2^-$	Ambient	O_2 , Cl_2 , ClO_2 , FClO_3 , KPF_6	8
$\text{PO}_2(\text{OH})$	Ambient	O_2 , ClO_2 , FClO_2 , PF_5 , POF_3	8
$\text{HPO}(\text{OH})_2$	Ambient	O_2 , Cl_2 , POF_3	8
$\text{Ce}^{+4}[\text{SO}_3(\text{OH})]_4^-$	Ambient	O_2 , ClO_2 , SO_2F_2 , SF_6 , solids	8
$\text{SO}_2(\text{OH})_2$	Ambient	O_2 , ClO_2 , SO_2F_2 , SOF_4	8
$\text{ClO}_3(\text{OH})$ (72 percent)	Ambient	O_2 , Cl_2 , ClO_2 , FClO_3	8
<u>Salts of Oxygen Acids</u>			
Na_2CO_3	Ambient	No reaction	8
	100 C	Reaction: O_2 , Cl_2 , CF_4 , CF_3Cl , NaF	8

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TABLE 5
(Continued)

Material	Test Temperature	Results and Products	Reference Number
<u>Salts of Oxygen Acids</u>			
NaNO_2	Ambient	Reaction: NO_2 , FNO , FNO_2 , FCIO_2 , Cl_2 , NaF	8
NaNO_3	Ambient	Reaction: FNO_2 , FCIO_2	8
$\text{Ca}_3(\text{PO}_4)_2$	Ambient	Reaction: O_2 , Cl_2 , ClO_2 , CaF_2 , $\text{Ca}(\text{PF}_6)_2$	8
$\text{Na}_2\text{S}_2\text{O}_3$	Ambient	No reaction	8
	150 C	Reaction: O_2 , Cl_2 , ClO_2 , SO_2F_2 , SF_6 , NaF	8
Na_2SO_3	Ambient	No reaction	8
	150 C	Reaction: O_2 , Cl_2 , ClO_2 , SO_2F_2 , SF_6 , NaF	8
KNO_3	-78 C	No reaction	17
K_2SO_4	Ambient	No reaction	8
	130 C	Reaction: O_2 , ClO_2 , SO_2F_2 , SF_6 , KF , KClF_4	8
KClO_3	-78 to -27 C	Limited reactions: FCIO_2	17
	Ambient	Reaction: O_2 , ClO_2 , FCIO_2 , FCIO_3 , KClF_4	8
KClO_4	-78 to -27 C	Limited reaction: FCIO_2	17
	Ambient; 150 C	No reaction	8
K_2CrO_4	Ambient	Slight reaction	8
	150 C	Reaction: O_2 , ClO_2 , FCIO_2 , FCIO_3 , CrO_2F_2 , KClF_4	8
$\text{K}_2\text{Cr}_2\text{O}_7$	Ambient	Reaction: O_2 , FCIO_3 , CrO_2F_2 , KClF_4	8

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TABLE 5
(Continued)

Material	Test Temperature	Results and Products	Reference Number
<u>Salts of Oxygen Acids</u>			
KMnO_4	Ambient	Reaction: O_2 , FCIO_2 , FMnO_3 , KF , KClF_4	8
CsNO_3	-78 to -25 C	Slow reaction	17
	25 C	Reaction completed after 3 days: NO_2F , ClO_2F	17
LiClO_4	-78 C	Reaction: LiF	23, 24
<u>Lewis Acids and Bases</u>			
KF	Ambient	No reaction	8
	150 C	No reaction	8
CsF	-78 C to ambient	No reaction	28, 8
	150 C	No reaction	8
TiF_4	Ambient	No reaction	8
BF_3	-112 C	No reaction	8
	Ambient	Reaction	18
	Ambient	No reaction	27
PF_5	0 C	No reaction	8
AsF_5	0 C	Reaction: $\text{ClF}_4^+ \text{AsF}_6^-$	8
	Ambient	Reaction: $\text{ClF}_4^+ \text{AsF}_6^-$	18 - 24
SbF_5	0 C	Reaction: $\text{ClF}_4^+ \text{SbF}_6^- \cdot \text{LSbF}_5$	8
	375 C	Reaction: $\text{ClF}_4^+ \text{SbF}_6^-$	27
SF_4	Ambient	No reaction	18, 27
SF_6	Ambient	No reaction	27

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TABLE 5
(Continued)

Material	Test Temperature	Results and Products	Reference Number
<u>Halides and Pseudohalides</u>			
HCl	Ambient	Reaction: HF, Cl ₂	8
NaCl	Ambient	Reaction: Cl ₂ , ClF, NaF	8
KI	Ambient	Reaction: Cl ₂ , KICl ₄ , KIF ₆	8
	150 C	Reaction: Cl ₂ , IF ₅ , KICl ₄ , KIF ₆	8
KCN	Ambient	Vigorous reaction: Cl ₂ , CF ₄ , N ₂ , C, KF	8
Hg(CN) ₂	Ambient	Vigorous reaction: Cl ₂ , ClF, CF ₄ , N ₂ , N ₂ F ₂ , HgF ₂	8
KOCN	Ambient	Reaction: O ₂ , Cl ₂ , N ₂ , CF ₄ , KF, KClF ₄	8
NaN ₃	-150 C	Violent reaction: Cl ₂ , NaF, N ₂	8
<u>Borides, Carbides, Nitrides, Sulfides and Azides</u>			
CaB ₆ (technical grade)	Ambient	Reaction: Cl ₂ , BF ₃ , O ₂ , CaF ₂	8
CaC ₂	Ambient	Slight reaction: CF ₄	8
	150 C	Reaction: Cl ₂ , CF ₄ , CF ₃ Cl, C, CaF ₂	8
B ₄ C	Ambient	No reaction	8
	150 C	Slight reaction: trace CF ₄	8

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TABLE 5
(Continued)

Material	Test Temperature	Results and Products	Reference Number
<u>Borides,</u> <u>Carbides,</u> <u>Nitrides,</u> <u>Sulfides</u> <u>and Azides</u>			
Al C ₄ (impure sample)	Ambient	Partial reaction: ClO ₂ , FCIO ₃ , CF ₄ , (appearance unchanged)	8
SiC	Ambient 150 C	No reaction Slight reaction: trace CF ₄	8 9
WC	Ambient 150 C	No reaction Reaction: Cl ₂ , CF ₄ , CF ₃ Cl, WF ₆	
Mg ₃ N ₂	Ambient 100 C	No reaction Reaction: ClNO ₂ , Cl ₂ , N ₂ , MgF ₂ , FNO ₂	8 8
Ca ₃ N ₂	Ambient	Reaction: ClNO ₂ , Cl ₂ , N ₂ CaF ₂	8
ZrN	Ambient 100 C	No reaction ClNO ₂ , Cl ₂ , N ₂ , ZrF ₄	8 8
CrN	Ambient 150 C	No reaction Partial reaction: FNO ₂ , Cl ₂ , N ₂ , CrF ₃	8 8
BN	Ambient	Reaction: Cl ₂ , N ₂ , BF ₃	8
Na ₂ S (techni- cal grade)	Ambient	Reaction: Cl ₂ , SF ₆ , SOF ₂ , NaF	8

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TABLE 5
(Continued)

Material	Test Temperature	Results and Products	Reference Number
ZnS	Ambient	Reaction: Cl_2 , SF_6 , SOF_2 , ZnF_2	8
COS	Ambient	Reaction: Cl_2 , COF_2 , COFCl , SF_4 , SOF_2	8
Azides (general)	-78 C to Ambient	Vigorous reaction	17
<u>Solvents</u>			
$(\text{CF}_3)_2\text{CO}$	-80 C	No reaction	8
	0 C	Violent reaction: O_2 , Cl_2 , COF_2 , CF_4	8
$(\text{CF}_3\text{CO})_2\text{O}$	-60 to -30 C	Slight solubility	8
CHCl_3	Ambient	Slow reaction: CHCl_2F , CCl_2F_2 , CFCl_3 , CF_4 , Cl_2	8
CCl_4	Ambient	Slow reaction: CFCl_3 , Cl_2	8
CFCl_3 (Freon 11)	Ambient	No reaction: no F^{19} exchange	8
CF_2Cl_2	-93 C	Forms homogeneous mixture	16
(Freon 12)	Ambient	Progressive fluorination of CCl_2F_2 after 10 days	32
CClF_3	-93 C	Forms homogeneous mixture	16
CF_4 (Freon 14)	-128 C	Solubility of solid ClF_5 <0.5 percent	16
$\text{CF}_2\text{ClCFCl}_2$ (Freon 113)	-30 C	Immiscible	8

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TABLE 5
(Concluded)

Material	Test Temperature	Results and Products	Reference Number
Fluorolube Oil	Ambient	No reaction: no F^{19} exchange	8
$(C_4F_9)_3N$ (N-43)	-30 C	Immiscible	8
	Ambient	No reaction: no F^{19} exchange, Miscible at 50 m/o	8
Kel F Oil (No. 3)	Ambient	Readily soluble	16
FC-75	Ambient	No reaction; no F^{19} exchange; miscible at 50 m/o	8
FC-77	Ambient	No reaction: no F^{19} exchange; miscible at 50 m/o	8
HF	-38 C	No reaction: 6 m/o solubility; no F^{19} exchange: solvent shift = 2.6 ppm downfield	8
	0 C	No reaction: >13 m/o solubility	8
	28 C	No reaction: no F^{19} exchange	8

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2.4 CHLORINE PENTAFLUORIDE MIXTURES

A number of propellant mixtures and new propellant concepts involving ClF_5 have been investigated under programs designed to formulate new and useful high-energy, earth-storable oxidizer mixtures. Organizations that have conducted experimental investigations of various homogeneous mixtures with ClF_5 , include Aerojet-General Corporation (Ref. 33 through 39), Allied Chemical Corporation (Ref. 18), Monsanto Research Corporation (Ref. 17), Reaction Motors Division of Thiokol Chemical Corporation (Ref. 7 and 40 through 43), and Rocketdyne (Ref. 8 and 45). In addition, Reaction Motors (Ref. 43 and 44) has also investigated heterogeneous mixtures with ClF_5 . For the most part, these investigations consisted of studies of formulation compatibilities, miscibilities, homogeneity, and/or engineering properties. Because these studies are related to potential application of ClF_5 , brief summations of the various formulations are presented in the following paragraphs.

2.4.1 ClF_5 - ClF_3 Mixtures

A ClF_5 - ClF_3 mixture (64 w/o ClF_3) was found to be miscible, with vapor pressure data demonstrating a slight positive deviation from ideality at ambient temperature (Ref. 17).

2.4.2 ClF_5 - BrF_5 Mixtures

A study (Ref. 17) of two ClF_5 - BrF_5 mixtures (42 and 62 w/o ClF_5) indicated miscibility and positive vapor pressure deviations from ideality. The deviations were more positive with increased ClF_5 content.

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2.4.3 ClF₅-FC10₃ Mixtures

Mixtures of ClF₅-FC10₃ were found to be completely miscible, compatible, and insensitive to shock (Ref. 7). The vapor pressures of various mixtures were reported (Ref. 8) to have slight positive deviations from ideality over a temperature range of -46 to 20 C; very little vapor pressure deviation was noted (Ref. 7) in an 80 w/o ClF₅-20 w/o FC10₃ mixture from -23 to 25 C.

2.4.4 ClF₅-XeF₂ Mixtures

The solubility of XeF₂ in ClF₅, determined over a temperature range of 0.1 to 41.2 C, ranged from 4.68 to 13.3 gm/100 gm ClF₅. This low solubility limited future use of this theoretically potential high-density impulse formulation (Ref. 45).

2.4.5 ClF₅-XeF₄ Mixtures

Experimental determinations of XeF₄ solubility in ClF₅ indicated that low solubilities (2.92 to 7.92 gm/100 gm ClF₅ over a temperature range of 4.5 to 45.5 C) would prevent effective application of this oxidizer mixture (Ref. 45).

2.4.6 ClF₅-OF₂ Mixtures

Mixtures of OF₂ and ClF₅ were found to be miscible at -78 C, but demonstrated large positive deviations from ideality (Ref. 17).

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2.4.7 $\text{ClF}_5\text{-HNF}_2$ Mixtures

A 3 to 1 mixture of $\text{ClF}_5\text{-HNF}_2$ detonated within 1 minute after mixing at -120°C (Ref. 7). However, it was also noted (Ref. 46) that very pure ClF_5 and HNF_2 reacted smoothly at -80°C to form ClNF_2 in good yields.

2.4.8 $\text{ClF}_5\text{-NF}_3$ Mixtures

Equal weights of ClF_5 and NF_3 were found to be miscible at -78°C in glass capillaries (Ref. 17).

2.4.9 $\text{ClF}_5\text{-N}_2\text{F}_4$ Mixtures

$\text{ClF}_5\text{-N}_2\text{F}_4$ mixtures have been extensively studied by Aerojet (Ref. 33 through 39), Reaction Motors (Ref. 7 and 41 through 43) and Rocketdyne (Ref. 8 and 45) as a primary candidate high-energy storable oxidizer formulation. Early studies (Ref. 7, 8 and 34) indicated reaction between the two compounds above ambient temperatures. Additional laboratory investigations (Ref. 35 through 45) confirmed decomposition of the compounds in various materials over a wide temperature range and suggested that decomposition was both homogeneous and heterogeneous in nature (Ref. 45).

Vapor pressure studies (Ref. 7, 8, 33, and 41) indicated slight positive deviations from ideality for various mixtures at temperatures from -46 to 71°C . Density determinations (Ref. 7 and 33) resulted in experimental values slightly higher than

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theoretical values based on additivity of molar volumes. Various laboratory sensitivity studies demonstrated the insensitive characteristics of various mixtures in relation to shock (Ref. 7), rapid compression (Ref. 34), and gaseous nitrogen pressurization (Ref. 42).

2.4.10 $\text{ClF}_5\text{-NF}_3$ Mixtures

The $\text{ClF}_5\text{-NF}_3$ mixture was found to be compatible and miscible in all proportions (Ref. 8); however, results from vapor pressure measurements by various investigators were in conflict. Measurements on $\text{ClF}_5\text{-NF}_3$ mixtures at -82 and -97 C (Ref. 8) indicated large positive deviations from ideality; moderate positive deviations were reported at -78 C (Ref. 17) and room temperature (Ref. 7). In complete disagreement with these data, strong negative deviations were also reported (Ref. 18) for mixtures at temperatures up to 24 C. The discrepancies in these data have not been resolved.

2.4.11 $\text{ClF}_5\text{-N}_2\text{O}_4$ Mixtures

N_2O_4 was found to be incompatible with ClF_5 . Pressures in excess of 350 psia were recorded after 18 hours when a $\text{ClF}_5\text{-N}_2\text{O}_4$ mixture was sealed in a Kel-F tube at ambient temperatures. Mass spectrometer analysis of the vapor phase revealed 25 percent Cl_2 , 10 percent SiF_4 , 5 percent ClO_2 , 60 percent NO_2 , NO , N_2O , and NO_2^+ species and complete decomposition of ClF_5 in this phase (Ref. 7).

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2.4.12 $\text{ClF}_5\text{-C}(\text{NO}_2)_4$ Mixtures

A preliminary experimental characterization of the $\text{ClF}_5\text{-C}(\text{NO}_2)_4$ system demonstrated compatibility between two compounds. Vapor pressure measurements at 0 and 20 C resulted in data very near ideality; however, mixtures containing more than 85 percent $\text{C}(\text{NO}_2)_4$ were solid at 0 C (Ref. 8).

2.4.13 $\text{ClF}_5\text{-FC}(\text{NO}_2)_3$ Mixtures

The compatibility and miscibility of $\text{ClF}_5\text{-FC}(\text{NO}_2)_3$ was verified over a ClF_5 composition range of 40 to 90 w/o. Vapor pressure measurements indicate high concentrations of ClF_5 form essentially ideal mixtures; however, mixtures with low concentrations of ClF_5 exhibit positive deviations from ideality indicating limited solubility of ClF_5 in $\text{FC}(\text{NO}_2)_3$. Mixtures containing equivalent amounts of ClF_5 and $\text{FC}(\text{NO}_2)_3$ were found to be more sensitive than the individual components (Ref. 7).

2.4.14 $\text{ClF}_5\text{-FC}(\text{NF}_2)_3$ Mixtures

Binary mixtures of ClF_5 and $\text{FC}(\text{NF}_2)_3$ were found compatible at various temperatures to 71 C (Ref. 7, 8, and 17). Although one investigator (Ref. 7) reported the apparent immiscibility of a 50-50 mixture at 0 C with solubility increasing as temperature increases, other investigators (Ref. 8 and 17) found miscible mixtures (including 50 percent mixtures) from -78 to 20 C and slight positive deviations in vapor pressure.

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2.4.15 $\text{ClF}_5\text{-C}(\text{NF}_2)_4$ Mixtures

Mixture screening studies from room temperature (Ref. 40) to 71 C (Ref. 8 and 45) indicate that ClF_5 is compatible and miscible with $\text{C}(\text{NF}_2)_4$. Vapor pressure measurements (Ref. 45) indicate slight positive deviations from 0 to 71 C. Trauzl block sensitivity tests (Ref. 40) gave positive results.

2.4.16 $\text{ClF}_5\text{-OF}_2\text{-ClF}_3$ Mixtures

Mixtures of $\text{ClF}_5\text{-ClF}_3\text{-OF}_2$ containing OF_2 concentrations of 2 and 7 w/o in the liquid phase at -78 C showed large positive deviations from ideality during vapor pressure measurements (Ref. 17).

2.4.17 $\text{ClF}_5\text{-OF}_2\text{-BrF}_5$ Mixtures

Vapor pressure measurements on two $\text{ClF}_5\text{-OF}_2\text{-BrF}_5$ mixtures at -78 C demonstrated large positive deviations from ideality (Ref. 17).

2.4.18 $\text{ClF}_5\text{-OF}_2\text{-NF}_3$ Mixtures

A ternary mixture of 37 w/o ClF_5 -14 w/o OF_2 -39 w/o NF_3 exhibited a large positive deviation in vapor pressure measurements at -78 C (Ref. 17).

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2.4.19 $\text{ClF}_5\text{-OF}_2\text{-CF(NF}_2)_3$ Mixtures

Ternary mixtures of $\text{ClF}_5\text{-OF}_2\text{-CF(NF}_2)_3$ exhibited large positive deviations in vapor pressure measurements at -78°C (Ref. 17).

2.4.20 $\text{ClF}_5\text{-OF}_2\text{-N}_2\text{F}_4$ Mixtures

A $\text{ClF}_5\text{-OF}_2\text{-N}_2\text{F}_4$ mixture of approximately equal parts was found to be miscible at -78°C in sealed Pyrex glass capillaries (Ref. 17).

2.4.21 $\text{ClF}_5\text{-FClO}_3\text{-N}_2\text{F}_4$ Mixtures

A study of the $\text{ClF}_5\text{-FClO}_3\text{-N}_2\text{F}_4$ ternary system demonstrated the compatibility, miscibility, and insensitivity of the system. Vapor pressure measurements on five different mixtures over a range of -45°C to ambient temperature showed very slight deviations from ideality (Ref. 7).

2.4.22 $\text{ClF}_5\text{-FClO}_3\text{-BrF}_5$ Mixtures

The homogeneity, vapor pressure, and storability studies were conducted on several mixtures of ClF_5 , FClO_3 , and BrF_5 (Ref. 47). A 52 w/o ClF_5 -11 w/o FClO_3 -37 w/o BrF_5 mixture was homogeneous at -76°C and demonstrated nearly ideal behavior with respect to vapor pressures to 165°F . Positive deviations were found in vapor pressures of a 69 w/o ClF_5 -11 w/o FClO_3 -20 w/o BrF_5 mixture during high temperature storage tests at 217°F ; however, posttest

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analysis indicated this was caused by the original presence of a noncondensable gas. This particular mixture was completely stable in type 347 CRES over a period of 22 weeks at this temperature.

2.4.23 $\text{ClF}_5\text{-N}_2\text{F}_4\text{-C}(\text{NO}_2)_4$ Mixtures

Reactions, noted in $\text{ClF}_5\text{-N}_2\text{F}_4\text{-C}(\text{NO}_2)_4$ ternary mixtures, have been attributed to reaction between N_2F_4 and ClF_5 . Homogeneity data indicate positive deviations in vapor pressures over almost all compositions and temperatures (0, 45, and 71 C); the deviation increases with increasing $\text{C}(\text{NO}_2)_4$ content and decreases with increasing temperature (Ref. 8).

2.4.24 $\text{ClF}_5\text{-N}_2\text{F}_4\text{-FC}(\text{NF}_2)_3$ Mixtures

Some homogeneity data have been generated for $\text{ClF}_5\text{-N}_2\text{F}_4\text{-FC}(\text{NF}_2)_3$ mixtures at low temperatures; however, potential incompatibility and sensitivity problems have eliminated interest in these mixtures (Ref. 8).

2.4.25 $\text{ClF}_5\text{-FC}(\text{NF}_2)_3\text{-C}(\text{NO}_2)_4$ Mixtures

The ternary system, $\text{ClF}_5\text{-FC}(\text{NF}_2)_3\text{-C}(\text{NO}_2)_4$, was found to be compatible and stable at 71 C. Data obtained at 0, 45, and 71 C demonstrated a positive deviation from ideality for all compositions and temperatures except for a negative deviation in a $\text{FC}(\text{NF}_2)_3$ -rich mixture at 71 C. The amount of positive deviation increased with increasing $\text{C}(\text{NO}_2)_4$ content and decreased with increasing temperature (Ref. 8). Sensitivity problems have also eliminated interest in these mixtures.

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2.4.26 $\text{ClF}_5\text{-C}(\text{NF}_2)_4\text{-C}(\text{NO}_2)_4$ Mixtures

The compatibility of the ternary system, $\text{ClF}_5\text{-C}(\text{NF}_2)_4\text{-C}(\text{NO}_2)_4$, was examined in type 304 stainless steel at 71 C. The composition of two mixtures, 68.5-5.7-25.8 and 68.9-7.5-23.6 w/o $\text{ClF}_5\text{-C}(\text{NF}_2)_4\text{-C}(\text{NO}_2)_4$, remained unchanged over a test period of 5 days (Ref. 45). Interest in these mixtures has been curtailed by sensitivity problems.

2.4.27 $\text{ClF}_5\text{-B}_4\text{C}$ Mixtures

Heterogeneous mixtures of ClF_5 and B_4C have been investigated in an effort to develop high-energy heterogeneous monopropellant systems (Ref. 43 and 44). Preliminary thermal stability and card gap sensitivity tests on $\text{ClF}_5\text{-B}_4\text{C}$ mixtures indicated possible stability and sensitivity problems.

2.5 CHLORINE PENTAFLUORIDE GELS

A variety of compounds have been investigated as potential gelling agents for ClF_5 (Ref. 17, 35 through 37, 48 and 49). Actual ClF_5 gels have been formed through use of 30 w/o calcium fluoride or 9.9 w/o precipitated lithium fluoride (Ref. 48), and 10 percent $\text{Ba}(\text{SbF}_6)_2$ (Ref. 37). In addition, blends of ClF_5 and ClF_3 (≤ 50 percent ClF_5) were gelled with $\text{Ba}(\text{SbF}_6)_2$ (Ref. 49).

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SECTION 3: MATERIALS OF CONSTRUCTION

3.1 MATERIALS COMPATIBILITY

The compatibility of ClF_5 with selected materials has been experimentally determined under various conditions associated with common utilization of materials in the fabrication and construction of propellant handling equipment and facilities. The primary data presented in this section are a result of studies (Ref. 3, 6, 9, 13, 16, and 50 through 52), that were, at least partially directed at the examination of materials compatibility with ClF_5 . The results of these studies, which were designed to provide basic information for the fabrication of test systems as well as to form a basis for more sophisticated efforts, are presented in Table 6. As a supplement to these data, various results of the ClF_5 chemistry studies (Table 5) and an experimental evaluation of selected Atlas Missile System components in ClF_5 service (Ref. 52) provide additional technology on materials compatibility.

The compatibilities of the various metals are rated in Table 6 from experimental data interpreted on the basis of practical experience. The test conditions are described and pertinent data are presented. Each individual metal was placed in one of four rating classifications corresponding to its compatibility under the exposure conditions described. These four rating classifications, which are similar to those used by the Defense Metals Information Center (Ref. 53), are as follows:

1. Metals suitable for unlimited use (after proper material preparation) under the specified exposure conditions. Corrosion rates are less than 1 mil per year (MPY). Typical use involves constant contact with ClF_5 .

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2. Metals suitable for transient and/or limited contact under the specified exposure conditions. Corrosion rates are less than 5 MPY. Typical uses are in hardware which handles the propellant intermittently.
3. Metals suitable for momentary contact. Corrosion rates are < 50 MPY. Prolonged or repeated exposures should be avoided.
4. Metals unsuitable for use under any condition. Corrosion rates are > 50 MPY.

The compatibility ratings for the nonmetallics are based on different standards but correspond on a usage basis with the metals classifications. Definition of the nonmetallic ratings are as follows:

1. Satisfactory for service under conditions indicated. No significant change is noted in the material or propellant.
2. Use with knowledge that the material will undergo slight changes in its physical properties. Slight changes may also occur in the propellant.
3. May be used where moderate chemical and physical degradation can be tolerated for a short period. Potential results from usage must be fully comprehended.
4. Unsatisfactory for use under conditions indicated. There is extensive or complete physical and/or chemical breakdown of the material or propellant.

A general summary of the results from ClF_5 materials compatibility studies indicates the behavior of ClF_5 with various structural materials is generally similar to that of ClF_3 . If the metallics are compatible under conditions of normal exposure to liquid and gaseous ClF_5 . Notable exceptions in the metals tested are molybdenum and columbium (niobium), which

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undergo complete reaction. Although tested, the compatibility of gold and titanium with ClF_5 is still in doubt because of conflicting results in comparison with ClF_3 . In addition, the presence of moisture may have a significant detrimental effect (Ref. 6) on the rate of film formation or corrosion of the metal. Of the metals tested under conditions of moisture contamination, only Hastelloy C and nickel 200 provided complete resistance to attack.

The number and types of compatible nonmetallic materials are limited with ClF_5 . Experimental efforts have shown that Kel-F and Teflon plastics, various fluorocarbon oils, and some noted specialty items are compatible under limited conditions (static application over ambient temperature ranges). Because very little useful data prescribing the actual limitations of non-metallic use with ClF_5 have been generated, proposed applications should be experimentally and thoroughly investigated. However, it should be noted that, based on the chemical similarity between ClF_5 and ClF_3 and the demonstrated compatibility of materials with ClF_3 , the compatibility of any nonmetallic with ClF_5 under dynamic conditions is highly suspect.

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TABLe 6

COMPATIBILITY OF MATERIALS WITH CIF⁵

Material	Exposure				Rating	Remarks	Reference Number		
	Type	Phase	Temperature, F	Time, Days					
<u>Aluminum Alloys</u> 1100	Static	Liquid	60 to 15	30	A	Slight weight gain	6		
			80 to 100	580	A	Slight weight loss	9		
			160	30	A	Slight weight gain	6		
	Vapor		60 to 75	30	A	Slight weight gain	6		
			30 to 100	580	A	Slight weight loss	9		
			160	30	A	Slight weight gain	6		
Static	Liquid	86	21	A	Slight weight change	50			
		2014-T6	Static	Liquid	152	21	A	No weight change	50
					86	-	A	~ 65 ft lb impact test	50
2021	Dynamic		1125 (metal)	-	A	Impingement of vapor on heated metal	51		
			1185 (metal)	-	B		51		
2024-T3	Static	Liquid	60 to 75	30	A	Slight weight loss	6		
			30 to 100	580	A	Slight weight loss	9		
			160	30	A	Slight weight gain	6		
	Vapor		Ambient	7	A	Slight weight gain	52		
			60 to 75	30	A	Slight weight loss	6		
			30 to 100	580	A	Slight weight loss	9		
		160	30	A	Slight weight gain	6			

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TABLE 6
(Continued)

Material	Exposure			Rating	Remarks	Reference Number
	Type	Phase	Temperature, F	Time, Days		
<u>Aluminum Alloys</u> (continued) 2024-T351	Static	Liquid	60 to 75	30	Slight weight loss	6
			30 to 100	580	Slight weight loss	9
			160	30	Slight weight gain	6
	Vapor		60 to 75	30	Slight weight loss	6
			30 to 100	580	Slight weight loss	9
			160	30	Slight weight loss	6
2219-T351	Static	Liquid	60 to 75	30	Slight weight loss	6
			30 to 100	580	Slight weight loss	9
			160	30	Slight weight gain	6
	Vapor		60 to 75	30	Slight weight loss	6
			30 to 100	580	Slight weight loss	9
			160	30	Slight weight gain	6
5086	Static	Liquid	60 to 75	30	No weight change	6
			30 to 100	580	Slight weight loss	9
			160	30	Slight weight gain	6
	Vapor		60 to 75	30	No weight change	6
			30 to 100	580	Slight weight loss	9
			160	30	Slight weight gain	6
5457	Static	Liquid	60 to 75	30	Slight weight loss	6
			30 to 100	580	Slight weight loss	9
			160	30	Slight weight gain	6
	Vapor		60 to 75	30	No weight change	6
			30 to 100	580	Slight weight loss	9
			160	30	Slight weight gain	6

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TABLE C
(Continued)

Material	Exposure			Rating	Remarks	Reference Number
	Type	Phase	Temperature, F	Time, Days		
<u>Aluminum Alloys</u> (continued) 5457		Vapor	60 to 75 30 to 100 160	30 580 30	Slight weight loss Slight weight loss Slight weight gain	6 9 6
	Static	Vapor	Ambient	7	Slight weight gain	52
	Static	Liquid	86 152	21 21	Slight weight change Slight weight change	50 50
6061-T651	Static	Liquid	60 to 75 30 to 100 160	30 580 30	Slight weight loss Slight weight loss Slight weight gain	6 9 6
		Vapor	60 to 75 30 to 100 160	30 580 30	Slight weight loss Slight weight loss Slight weight gain	6 9 6
	Static	Liquid	60 to 75 30 to 100 160	30 580 30	Slight weight loss Slight weight loss Slight weight gain	6 9 6
6066-T6	Static	Liquid	60 to 75 30 to 100 160	30 580 30	Slight weight loss Slight weight loss Slight weight gain	6 9 6
		Vapor	60 to 75 30 to 100 160	30 580 30	Slight weight loss Slight weight loss Slight weight gain	6 9 6
	Static	Liquid	60 to 75 30 to 100 160	30 580 30	Slight weight loss Slight weight loss Slight weight gain	6 9 6

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TABLE 6
(Continued)

Material	Exposure			Rating	Remarks	Reference Number
	Type	Phase	Temperature, F	Time, Days		
<u>Aluminum Alloys</u> (continued) X7002	Static	Liquid	60 to 75	30	Slight weight gain	6
			30 to 100	580	Slight weight loss	9
			160	30	Slight weight gain	6
		Vapor	60 to 75	30	Slight weight gain	6
			30 to 100	580	Slight weight loss	9
			160	30	Slight weight gain	6
7075-T6	Static	Liquid	60 to 75	30	Slight weight gain	6
			30 to 100	580	Slight weight loss	9
			160	30	Slight weight gain	6
		Vapor	60 to 75	30	Slight weight gain	6
			30 to 100	580	Slight weight loss	9
			160	30	Slight weight gain	6
7079-T651	Static	Liquid	60 to 75	30	Slight weight gain	6
			30 to 100	580	Slight weight loss	9
			160	30	Slight weight gain	6
		Vapor	60 to 75	30	Slight weight gain	6
			30 to 100	580	Slight weight loss	9
			160	30	Slight weight gain	6
<u>Austenitic Stainless Steels</u> 304	Static	Liquid	60 to 75	30	Slight weight gain	6
			30 to 100	580	Slight weight gain	9
			160	30	Slight weight gain	6

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TABLE 6
(Continued)

Material	Exposure			Rating	Remarks	Reference Number
	Type	Phase	Temperature, F	Time, Days		
Austenitic Stainless Steels (Continued)	Static	Vapor	60 to 75	30	Slight weight gain	6
			30 to 100	580	Slight weight gain	9
			160	30	Slight weight gain	6
304	Dynamic		1078 (metal)	-	Impingement of vapor on heated metal	51
			1502 (metal)	-		51
316	Static	Liquid	60 to 75	30	Slight weight gain	6
			30 to 100	580	Slight weight gain	9
			160	30	Slight weight gain	6
		Vapor	60 to 75	30	Slight weight gain	6
			30 to 100	580	Slight weight gain	9
			160	30	Slight weight gain	6
347	Static	Liquid	60 to 75	30	Slight weight gain	6
			86	21	No weight change	50
			30 to 100	580	Slight weight gain	9
			152	21	No weight change	50
			160	30	Slight weight gain	6
		Vapor	60 to 75	30	Slight weight gain	6
			30 to 100	580	Slight weight gain	9
			160	30	Slight weight gain	6

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TABLE 6
(Continued)

Material	Exposure			Rating	Remarks	Reference Number
	Type	Phase	Temperature, F	Time, Days		
<u>Austenitic Stainless Steels</u> (continued)	Dynamic	Liquid	80	-	~ 65 ft lb impact test	50
		Vapor	1125 (metal) 1150 (metal)	-	Impingement of vapor on heated metals	51 51
<u>Non-austenitic Stainless Steel</u> PH15-7 Mo	Static	Liquid	60 to 75 30 to 100 160	30 580 30	Slight weight gain Slight weight gain Slight weight gain	6 9 6
		Vapor	60 to 75 30 to 100 160	30 580 30	Slight weight gain No weight change Slight weight gain	6 9 6
		Liquid	167	175	Slight weight loss	15
	Static	Liquid	60 to 75 30 to 100 160	30 580 30	Slight weight gain Slight weight gain Slight weight gain	6 9 6
		Vapor	60 to 75 30 to 100 160	30 580 30	Slight weight loss Slight weight gain Slight weight gain	6 9 6
		Liquid	167	175	Slight weight loss	15
AM 347	Static	Liquid	167	175	Slight weight loss	15
AM 350	Static	Liquid	60 to 75 30 to 100 160	30 580 30	Slight weight gain Slight weight gain Slight weight gain	6 9 6
		Vapor	60 to 75 30 to 100 160	30 580 30	Slight weight loss Slight weight gain Slight weight gain	6 9 6
		Liquid	167	175	Slight weight loss	15

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TABLE 6
(Continued)

Material	Exposure			Rating	Remarks	Reference Number
	Type	Phase	Temperature, F	Time, Days		
<u>Non-austenitic Stainless Steels</u> (continued)						
	Static	Liquid and Vapor*	96	30	Metallographic studies indicated definite attack	6
AM 355	Static	Liquid	60 to 75	30	Slight weight gain	6
			30 to 100	580	Slight weight gain	9
			160	30	Slight weight gain	6
			167	175	Slight weight gain	13
	Vapor		60 to 75	30	Slight weight gain	6
			30 to 100	580	Slight weight gain	9
410	Static	Liquid and Vapor*	160	30	Slight weight gain	6
			86	30	Metallographic studies indicated definite attack	6
			60 to 75	30	Moderate weight gain indicated; however, rate change data remain < 1 MPY	6
			30 to 100	21		50
	Vapor		152	21		9
			160	30		50
	Static	Liquid	60 to 75	30		6
			30 to 100	580		9
			160	30		6

*Moisture contaminated

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TABLE 6
(Continued)

Material	Exposure				Rating	Remarks	Reference Number
	Type	Phase	Temperature, F	Time, Days			
Nickel and Nickel-Base Alloys Nickel (99.99 percent)	Static	Vapor	Ambient	1	A	No obvious reaction	52
				30	A	Slight weight gain	6
				21	A	No weight change	50
				580	A	Slight weight gain	9
				21	A	Slight weight change	50
Nickel 200	Static	Liquid	60 to 75 86	30	A	Slight weight loss	6
				152	A	Slight weight gain	6
				160	A	Slight weight gain	9
				30	A	Slight weight loss	6
				30	A	Slight weight gain	6
Nickel 211	Static	Vapor	60 to 75 30 to 100 160	30	A	Slight weight gain	6
				580	A	Slight weight loss	9
				30	A	Slight weight loss	6
				30	A	Slight weight gain	6
				30	A	Slight weight gain; no attack noted in metallographic analysis	6
Nickel 211	Dynamic	Liquid	86	-	A	~ 65 ft lb impact test	50
				30	A	Slight weight gain	6
				580	A	Slight weight gain	9
				30	A	Slight weight gain	6
				30	A	Slight weight gain	6

*Moisture contaminated

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TABLE 6
(Continued)

Material	Exposure			Rating	Remarks	Reference Number
	Type	Phase	Temperature, F	Time, Days		
Nickel and Nickel-Base Alloys (continued) Nickel 211	Static	Vapor	60 to 75	30	Slight weight gain	6
			30 to 100	580	Slight weight loss	9
			160	30	No weight change	6
			86	30	Some surface attack noted; however, weight change negligible	6
Inconel X-750	Static	Liquid	60 to 75	30	Slight weight gain	6
			30 to 100	580	Slight weight gain	9
			160	30	No weight change	6
			60 to 75	30	Slight weight gain	6
Bene' 41	Static	Vapor	30 to 10	580	Slight weight gain	9
			160	30	No weight change	6
			86	30	Some attack noted	6
			60 to 75	30	Slight weight gain	6
Bene' 41	Static	Liquid	30 to 100	580	Slight weight gain	9
			160	30	Slight weight gain	6
			160	30	Slight weight gain	6

*Moisture contaminated

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TABLE 6
(Continued)

Material	Exposure			Rating	Remarks	Reference Number
	Type	Phase	Temperature, F	Time, Days		
<u>Nickel and Nickel-Base Alloys</u> (continued) Rene' 41	Static	Vapor	60 to 75	30	Slight weight gain	6
			30 to 100	580	Slight weight gain	9
			160	30	Slight weight gain	6
Hastelloy C	Static	Liquid	60 to 75	30	Slight weight gain	6
			30 to 100	580	Slight weight gain	9
			160	30	Slight weight gain	6
			60 to 75	30	Slight weight gain	6
			30 to 100	580	Slight weight loss	9
<u>Copper and Monel Alloys</u> Monel 400	Static	Vapor	160	30	No weight change	6
			86	31	No attack noted	6
			60 to 75	30	Slight weight gain	6
			86	21	No weight change	50
			30 to 100	580	Slight weight gain	9
	Static	Liquid	152	21	No weight change	50
			160	30	Slight weight gain	6

*Moisture contaminated

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TABLE 6
(Continued)

Material	Exposure			Rating	Remarks	Reference Number
	Type	Phase	Temperature, F	Time, Days		
<u>Copper and Monel Alloys (continued)</u> Monel 400	Static	Vapor	60 to 75	30	Slight weight gain	6
			30 to 100	580	Slight weight gain	9
			160	30	Slight weight gain	6
		Liquid and Vapor*	86	30	Increased attack noted in comparison to uncontaminated exposure	6
Monel 402	Static	Liquid	60 to 75	30	Slight weight gain	6
			30 to 100	580	Slight weight gain	9
			160	30	Slight weight loss	6
		Vapor	60 to 75	30	Slight weight gain	6
			30 to 100	580	Slight weight gain	9
			160	30	Slight weight loss	6
			86	30	Increased attack noted in comparison to uncontaminated exposure	6
		Liquid and Vapor*				
Monel R-405	Static	Liquid	60 to 75	30	Slight weight gain	6
			30 to 100	580	Slight weight gain	9
			160	30	Slight weight loss	6

*Moisture contaminated

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TABLE 6
(Continued)

Material	Exposure			Rating	Remarks	Reference Number
	Type	Phase	Temperature, F	Time, Days		
<u>Copper and Monel Alloys (continued)</u> Monel B-405	Static	Vapor	60 to 75	30	Slight weight gain	6
			30 to 100	580	Slight weight gain	9
			160	30	Slight weight gain	6
			86	30	Increased attack noted in comparison to uncontaminated exposure	6
Monel K-500	Static	Liquid	60 to 75	30	Slight weight gain	6
			30 to 100	580	Slight weight gain	9
			160	30	Slight weight gain	6
		Vapor	60 to 75	30	Slight weight gain	6
			30 to 100	580	Slight weight gain	9
			160	30	Slight weight gain	6
Monel 501	Static	Liquid	86	30	Increased attack noted in comparison to uncontaminated exposure	6
			60 to 75	30	Moderate weight gain	6
			30 to 100	580	Slight weight gain	9
			160	30	Slight weight gain	6

*Moisture contaminated

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TABLE 6
(Continued)

Material	Exposure			Rating	Remarks	Reference Number
	Type	Phase	Temperature, F	Time, Days		
Copper and Monel Alloys (continued) Monel 501	Static	Vapor	60 to 75	30	Moderate weight gain	6
			30 to 100	580	Slight weight gain	9
			160	30	Slight weight gain	6
			86	30	Increased attack noted in comparison to uncontaminated exposure	6
Monel 505	Static	Liquid	60 to 75	30	Slight weight gain	6
			30 to 100	580	Slight weight gain	9
			160	30	Slight weight loss	6
		Vapor	60 to 75	30	Slight weight gain	6
			30 to 100	580	Slight weight gain	9
			160	30	No weight change	6
Monel 507	Static	Liquid	86	30	Increased attack noted in comparison to uncontaminated exposure	6
			60 to 75	30	Slight weight gain	6
			30 to 100	580	Slight weight gain	9
			160	30	Moderate weight gain	6

*Moisture contaminated

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TABLE 6
(Continued)

Material	Exposure			Rating	Remarks	Reference Number
	Type	Phase	Temperature, F	Time, Days		
Copper and Monel Alloys (continued)	Static	Vapor	60 to 75	30	Slight weight gain	6
			50 to 100	580	Slight weight gain	9
			160	30	Slight weight gain	6
		Liquid and Vapor*	86	30	Heavy attack noted	6
ETP Copper	Static	Liquid	60 to 75	30	Slight weight gain	6
			86	81	Slight weight change	50
			30 to 100	580	Slight weight gain	9
		Vapor	152	21	Slight weight change	50
			160	30	Slight weight gain	6
			60 to 75	30	Slight weight gain	6
Copper (soft)	Static	Vapor	50 to 100	580	Slight weight gain	9
			160	30	Slight weight gain	6
			86	30	Increased attack noted in comparison to uncontaminated exposure	6
		Vapor	Ambient	7	Slight weight gain	52

*Moisture contaminated

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TABLE 6
(Continued)

Material	Exposure			Rating	Remarks	Reference Number
	Type	Phase	Temperature, F	Time, Days		
<u>Miscellaneous Metals</u>						
Yellow Brass, 1/2 Hard	Static	Liquid	86	21	Slight weight gain Increased weight gain; but < 1 MPY	50
			152	21		50
Columbium (Niobium)	Static	Liquid	86	< 1	Complete reaction (not violent)	50
Gold (99.99 percent)	Static	Vapor	Ambient	1	Reacted to form compound	52
Indium (99.99 percent)	Static	Vapor	Ambient	1	Some surface reaction	52
Lead (sheet)	Static	Vapor	Ambient	1	Weight increased 2 percent	52
Magnesium AZ51B	Static	Liquid	86	21	Slight weight change	50
			152	21		50
Molybdenum	Static	Liquid	86	-	~ 65 ft lb impact test	50
			86	< 1		50
Platinum (99.99 percent)	Static	Vapor	Ambient	1	Weight increased 2.5 percent	52
			Ambient	1		52

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TABLE 6
(Continued)

Material	Exposure			Rating	Remarks	Reference Number
	Type	Phase	Temperature, F	Time, Days		
Miscellaneous Metals (continued)						
Titanium	Static	Liquid	86	< 1	No apparent change	50
Armalon A (aluminum-coated Teflon)	Static	Vapor	Ambient	8 hours	Weight increased 4 percent; reaction with Al coating	52
Armalon G (gold-coated Teflon)	Static	Vapor	Ambient	8 hours	Weight increased 4 percent; no chemical reaction	52
Asbestos Cloth (Specification SS-C-466)	Static	Vapor	Ambient	30 seconds	Ignited	52
Asbestos Gasket Neo-prene filled (MIL-A-7021)	Static	Vapor	Ambient	30 seconds	Ignited	52
Buna O-Ring (MIL-5516)	Static	Vapor	Ambient	7	Weight increased 103 percent; exploded with friction and/or shock	52
Calcium Sulfate (Drierite)	Static	Vapor	Ambient	8 hours	Absorbed ClF ₅ with no visible reaction	52

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TABLE 6
(Continued)

Material	Exposure			Rating	Remarks	Reference Number
	Type	Phase	Temperature, F	Time, Days		
Miscellaneous Metals (continued)						
Carbon (spectroscopic grade)	Static	Liquid	86	< 1	No chemical attack; crumbled to powder	50
CCl ₄ -CHCl ₃ (constant temperature bath fluid)	Static	Liquid	Ambient	-	Potential violent reaction when ClF ₅ spilled in CCl ₄ -CHCl ₃ bath	6
Dirt (red)	Static	Vapor	Ambient	30 seconds	Reaction and fuming	52
Fluorocarbon-Black	Static	Vapor	Ambient	7	Weight increased 4 percent; negative results at 2 ft-lb shock	52
Fluorocarbon-Green	Static	Vapor	Ambient	7	Weight increased 6 percent; negative results at 2 ft-lb shock	50
Fluorocarbon-Gold	Static	Vapor	Ambient	7	Weight increased 5 percent; negative results at 2 ft-lb shock	50

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TABLE 6
(Continued)

Material	Exposure				Rating	Remarks	Reference Number
	Type	Phase	Temperature, F	Time, Days			
<u>Miscellaneous Metals (continued)</u>							
Glass-Pyrex (dry)	Static	Liquid	Ambient	3 hours	C	Reaction at glass surface; extensive reaction and ClO ₂ formation	13
		Liquid and Vapor		30 minutes	C		6
Glass-Quartz (dry)	Static	Liquid and Vapor	-55 to 300	Several	A	No reaction observed when glass and Cl ₂ is dry	6
Glass-Vycor (dry)	Static	Liquid and Vapor	Ambient	30 minutes	C	Extensive reaction and ClO ₂ formation	6
Fluorocarbon Oil (FS5)	Static	Vapor	Ambient	16 hours		Inconclusive results	52
Fluorocarbon Oil - FC-75 (constant temperature bath fluid)	Static	Liquid	Ambient		A	No apparent reaction when ClF ₃ spilled into FC-75 bath	6
Graphite (spectroscopic grade)	Static	Liquid	86	< 1	D	No chemical attack; crumbled to powder	50

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TABLE 6
(Continued)

Material	Exposure			Rating	Remarks	Reference Number
	Type	Phase	Temperature, F	Time, Days		
<u>Miscellaneous Metals (continued)</u>						
CCl ₄ (constant temperature bath fluid)	Static	Liquid	Ambient	-	Potential violent reaction when ClF ₅ spilled in CCl ₄ bath	6
Kel F (sheet)	Static	Liquid	86 Ambient	< 1 30 minutes	Moderate weight gain Some absorption of ClF ₅	50 3
		Liquid and Vapor	30 to 160	30 to 580	Some absorption of ClF ₅	6, 9
Kel F 81	Static	Liquid	30 to 100	580	Moderate weight gain	9
Kel F 300	Static	Liquid	Ambient	30 minutes	Some absorption of ClF ₅	3
Kel F Elastomer	Static	Liquid	Ambient	30 minutes	Swelled, frayed, and tacky	3
Kel F Gasket (AMS 3650)	Static	Vapor	Ambient	7	Decomposed, foamed, soft and powdery	52
Kel F Grease (Kel F 30)	Static	Liquid	Ambient Vapor	- 16 hours	Reacts readily Decomposed; became a light liquid	16 52

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TABLE 6
(Continued)

Material	Exposure			Rating	Remarks	Reference Number
	Type	Phase	Temperature, F	Time, Days		
Miscellaneous Metals (continued)						
Lithium Fluoride Window	Static	Vapor	Ambient	2	Potential use as spectrophotometric window	52
Mylar Sheet	Static	Vapor	Ambient	D	Decomposed	52
Sodium Chloride Window	Static	Vapor	Ambient	D	Window surface deposit rendered it useless as optical window	52
Teflon	Static	Liquid	Ambient	30 minutes	No apparent change	3
Teflon 100	Static	Liquid	Ambient	30 minutes	No apparent change	3
Teflon FEP	Static	Liquid and Vapor	30 to 160	30 to 580	Moderate weight gain	6, 9
Teflon TFE	Static	Liquid and Vapor	30 to 160	30 to 580	Moderate weight gain	6, 9
		Liquid	86	< 1	No apparent change	50

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TABLE 6
(Concluded)

Material	Exposure			Rating	Remarks	Reference Number
	Type	Phase	Temperature, F	Time, Days		
Miscellaneous Metals (continued)						
Teflon TFE	Dynamic	Liquid	86	-	Completely reacted when impacted by 410 GRES at ~ 65 ft-lb in ClF ₅	50
Teflon Gasket (0-75063)	Static	Vapor	Ambient	7	Moderate weight gain	52
Teflon Sheet	Static	Vapor	Ambient	7	Moderate weight gain	52
Teflon Tube	Static	Liquid	Ambient	30	Diffusion of liquid through tube	6
		Vapor	Ambient	~ 7	Diffusion of liquid through tube	52
Transite	Static	Vapor	Ambient	30 seconds	Ignited	52
Viton A (carbon filled)	Static	Liquid	Ambient	30 minutes	Swelled, partial breakdown	3
Viton A Gasket	Static	Vapor	Ambient	7	Melted and flowed during tests	52

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3.2

PREPARATION OF MATERIALS

All components of a chlorine pentafluoride handling and/or storage system must be properly prepared prior to installation. In addition, the assembled system must be carefully dried and passivated. These procedures render all surfaces to be exposed to the propellant chemically inert.

Items such as valves, pumps, etc., cannot be cleaned in the assembled state because it is very difficult to remove all contaminants that might be present. Consequently, the cleaning of these items must be accomplished before the component parts are assembled.

The preparation of materials generally consists of degreasing, descaling, passivating, and drying. The cleaning solutions utilized during these operations shall be applied by immersing, spraying, circulating, or any other manner, as long as the surfaces to be cleaned are completely wetted in the solutions. Any component which can trap or retain liquids shall be drained or emptied between applications of different cleaning solutions.

All solutions shall be made with distilled, deionized, or clean tap water and all chemicals shall be of chemically pure grade or better. The water shall be filtered through a 40-micron nominal-size filter.

3.2.1

Degreasing

Components fabricated of stainless steel, copper, and aluminum alloys can be degreased by cold flushing or vapor degreasing with trichloroethylene, or by flushing with a mild alkaline solution containing from 5 to 7 ounces of Turco #4090* (or

*Turco #4090 is furnished by Turco Products, Inc., 6135 So. Central Avenue, Los Angeles, California.

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equivalent) per gallon of water at 140 to 160 F. The application of the mild alkaline solution shall be followed by a thorough water rinse.

Nonmetallic components, such as valve-stem packings fabricated of Teflon, can be degreased by immersion or scrubbing with the mild alkaline solution previously described, followed by a thorough water rinse.

Items such as nonmetallic components or simple components fabricated of machine metal stock, which are not to be cleaned any further, shall be dried by flushing with dry, hydrocarbon-free, filtered nitrogen gas or by heating in an oven at 140 to 160 F.

3.2.2 Descaling

Newly fabricated or reworked components which have scale resulting from welding or heat treatment, or impurities resulting from casting or forging, shall be descaled. Descaling solutions should not be used after precision machining unless the finished surfaces are protected.

The descaling of stainless-steel components is accomplished as follows:

1. Etch at room temperature for a period of no longer than 60 minutes with an aqueous solution containing from 3 to 5 weight percent technical grade hydrofluoric acid and from 15 to 20 weight percent technical grade nitric acid.
2. Rinse thoroughly with water to remove all traces of the descaling solution.

NOTE: If the components are to be passivated immediately after descaling, they need not be dried. Otherwise, the components may be dried by purging with dry, hydrocarbon-free filtered nitrogen gas or by heating in an oven at 140 to 160 F.

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Components fabricated of copper can be descaled as follows:

1. Descal with an aqueous solution containing approximately 20 percent (by volume) technical grade hydrochloric acid, at room temperature, until the surfaces are bright and free of oxidation.
2. Rinse with water to remove all traces of the descaling solution.

NOTE: After being descaled, the components require no further chemical treatment. They should be dried by purging with dry, hydrocarbon-free filtered nitrogen gas or by heating in an oven at 140 to 160 F.

The descaling procedure for components fabricated of aluminum alloys is as follows:

1. Clean with Turco Smut-Go* solution (1 lb/gallon of water), or an approved equivalent cleaner, until the surfaces are visibly clean and shiny.
2. Rinse with water to remove all traces of the acid solution. If the components are to be passivated immediately after descaling, they need not be dried. Otherwise, the components may be dried by purging with dry, hydrocarbon-free, filtered nitrogen gas or by heating in an oven at 140 to 160 F.

3.2.3 Passivating

The passivation procedure for components fabricated of stainless steel is as follows:

1. Immerse for a minimum period of 30 minutes, at room temperature, in an aqueous solution containing from 45 to 55 percent (by weight) technical grade nitric acid.

*Turco Smut-Go is a chromic acid cleaner furnished by the Turco Products, Inc., 6135 So. Central Avenue, Los Angeles, California.

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2. Rinse with water to remove all traces of the passivating solution.
3. Dry by purging with dry, hydrocarbon-free, filtered nitrogen gas or by heating in an oven at 140 to 160 F.

NOTE: Acid passivation of components having polished or lapped surfaces may be omitted if the finished surfaces cannot be conveniently protected from the acid solution.

Components fabricated of aluminum alloys can be passivated as follows:

1. Immerse for a minimum period of 1 hour, at room temperature, in an aqueous solution containing approximately 45 percent (by weight) technical grade nitric acid.
2. Rinse thoroughly with water to remove all traces of the passivating solution.
3. Dry by purging with dry, hydrocarbon-free, filtered nitrogen gas, or by heating in an oven at 140 to 160 F.

3.2.4 Handling

Items that have been prepared for chlorine pentafluoride service shall be handled, stored, or packaged in a manner to prevent recontamination. Large components such as valves, piping sections, tanks, etc., should have all openings capped with clean, compatible materials. Small items can be sealed in clean plastic bags.

3.2.5 System Passivation

After the chlorine pentafluoride system has been assembled and leak-checked, it is necessary to propellant-passivate the system. This is accomplished by introducing a small

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amount of chlorine pentafluoride vapor into the system. The gaseous chlorine pentafluoride not only reacts with any residual contaminating material, but also slowly attacks the containing metal surfaces forming an inert metal-fluoride film.

The passivation of chlorine pentafluoride systems can be accomplished as follows:

1. Evacuate the system by means of a high-capacity vacuum pump for at least 2 hours to remove any volatile contaminant or water vapor that might be present.
2. Place a slight positive pressure in the system using dry, hydrocarbon-free nitrogen gas.
3. Disconnect the vacuum pump, and cap and seal the open system connection.
4. Introduce gaseous chlorine pentafluoride slowly into the system until a pressure of approximately 20 to 25 psig is obtained.
5. After the gaseous chlorine pentafluoride has been in the system for approximately 10 minutes, bleed the system slowly to ambient pressure.
6. Introduce gaseous chlorine pentafluoride into the system until a pressure of approximately 20 to 25 psig is again obtained.
7. After the gaseous chlorine pentafluoride has been in the system for approximately 6 hours, bleed the system slowly to a pressure slightly above ambient.

After completion of the seven preceding steps, the system is considered passivated and ready to accept liquid chlorine pentafluoride. Precautions must be taken to prevent the entry of

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moisture into the system. This can be accomplished by using dry pressurizing gas and by maintaining a positive pressure in the system at all times.

NOTE: Although it is preferable to use gaseous ClF_5 as the passivating agent, it has been demonstrated that gaseous ClF_3 is effective as an alternative passivating material, if desired. Fluorine gas has also been used as a passivating agent; however, its effectiveness is apparently less than that of ClF_5 or ClF_3 .

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SECTION 4: HAZARDS

4.1 TOXICITY

4.1.1 Vapor Inhalation

Chlorine pentafluoride is a highly toxic compound and inhalation of even dilute concentrations must be avoided. Results of toxicological range studies with animals at Rocketdyne (Ref. 54) have shown a marked qualitative similarity of chlorine pentafluoride to chlorine trifluoride (Ref. 55 and 56) with respect to mode of attack and symptomatology. It was noted during these studies, however, that because of the apparent greater reactivity of chlorine pentafluoride with moisture in the air, the end amount of chlorine pentafluoride reaching the animal was considerably less than that of the chlorine trifluoride using the same initial concentrations.

Until more systematic and definitive studies have been conducted, the toxicity level presently utilized is that previously established for chlorine trifluoride (Ref. 57). This level is expressed as a threshold limit value (TLV) of 0.1 ppm. The TLV value represents the average concentration over a normal work day to which the average human can be safely exposed on a daily basis without adverse effects.

The odor threshold for chlorine pentafluoride vapors is very low, but has not been established reliably. However, personnel have detected the presence of chlorine pentafluoride vapors for short periods without suffering any apparent ill effects. Although the propellant odor has been used as a warning of potential danger with immediate personnel protective measures (evacuation of the hazard area and/or the use of self-contained or filtered breathing air) required, it is recommended that vapor detection devices be utilized in conjunction with an alarm system.

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If an individual is exposed to strong vapor concentrations of chlorine pentafluoride, he should hold his breath, if possible, until fresh air is reached or adequate breathing equipment is put on. If the person must breathe the vapor, breathing should be as shallow as possible. The exposed individual should be placed in the care of an authorized physician as soon as possible; in the meantime, first-aid treatment can be administered as directed by the local medical authority. For this purpose, it is recommended that personnel assigned to propellant handling and storage areas be properly trained in first-aid techniques. These techniques must be established only by the responsible medical authority.

Although symptoms of chlorine pentafluoride exposure have not been completely characterized, preliminary data indicate they are similar to those observed from chlorine trifluoride exposure. Thus, until additional toxicological studies have been conducted with ClF_5 , the data available on ClF_3 should be utilized as a guide. The symptoms of ClF_3 inhalation will vary according to vapor concentration, duration of exposure, and the individual. Exposure to olfactory detectable concentrations for short periods of time usually results in watering of the eyes, dry throat, chest pain, and sometimes coughing. Exposure to larger concentrations or prolonged exposure will result in gasping for breath, swelling of the eyes and eyelids, cloudiness of the cornea, lachrimation, severe salivation, coughing, breathing difficulty, and possibly convulsions or vomiting. In practice, fatal concentrations would be so irritating to the eyes and respiratory tract as to make the area intolerable. However, exposure to concentrations of 50 ppm or more of chlorine trifluoride for 15 to 30 minutes may result in subsequent death.

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4.1.2 Cutaneous Exposure

Chlorine pentafluoride is extremely corrosive and any contact of the propellant with living tissue will result in severe chemical burns. These deep, painful burns can cause serious tissue damage especially in the eyes. If a person has suffered skin or eye exposure to liquid or vaporized chlorine pentafluoride, the exposed areas should be washed immediately with large quantities of water for a period of at least 15 minutes. A continuous flow of water should not exceed 5 minutes and should be followed by a short rest interval. The affected individual should be placed in the care of an authorized physician as soon as possible.

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4.2 FIRE AND EXPLOSIVE HAZARDS

Preliminary detonation sensitivity tests (Ref. 3) indicate that ClF_5 is insensitive to initiation and will not propagate a detonation. Impact sensitivity tests with a modified JANAF drop-weight tester in dry nitrogen and dry air at liquid temperatures to 0 F showed no evidence of detonation up to 100 in.-lb, the limit of the tester. A modified cap-in-pipe test in 1/2-inch GRES tubing at 0 F resulted in no evidence of propagation of a detonation initiated by a 50-gram Compound C charge and blasting cap.

Additional tests (Ref. 6) with the U-tube apparatus demonstrated the insensitive nature of ClF_5 to adiabatic compression stimulus over a variety of selected "worst conditions" (both air and water contaminated). Two detonation propagation tests in 1-inch-OD GRES tubes provided further verification that liquid ClF_5 will not propagate a detonation.

The insensitive nature of ClF_5 was also confirmed by a standard Trauzl block test (Ref. 7).

The greatest fire and explosion hazard relating to the utilization of this oxidizer is related to its extreme reactivity with a variety of materials commonly found in propellant handling areas and systems. Although ClF_5 is nonflammable in air, its oxidizing potential is sufficiently strong to ignite and support combustion with fuels, many common materials of construction, solvents, organic contaminants and residues, etc. As indicated in the section on chemical properties, chlorine pentafluoride even reacts vigorously with water.

The energy release generated by these reactions is often large enough to initiate burning of other normally compatible materials (such as metal) with the oxidizer. Explosions involving chlorine pentafluoride are usually attributed to the rapid energy releases and attendant pressure buildup in confined systems.

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SECTION 5: SAFETY CRITERIA

5.1 HAZARD PREVENTION

As described in the Hazards Section, spills and leakage of chlorine pentafluoride can result in extreme hazards to both personnel and facilities. The best possible means of avoiding these hazards is elimination or minimization of the potential cause factors. Effective reduction of leakage, spills, and other potentially hazardous situations can be best accomplished by the use of properly designed equipment and thoroughly trained personnel.

5.1.1 System Integrity

The importance of the design integrity of the propellant's storage, transfer, and handling system cannot be overemphasized. The systems should be reliable, operationally flexible, and easy to maintain. Some of the suggested design criteria that should be incorporated in the system are as follows:

1. Only materials of construction which are definitely known to be compatible with the oxidizer shall be employed.
2. The system shall be designed and operated in such a manner as to prevent contamination of the system with moisture and any other known reactive materials.
3. The number of mechanical joints shall be reduced to a minimum, thus reducing the probability of propellant leakage.

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4. The system shall be designed to safely withstand the maximum operating pressure.
5. The transfer lines shall be free of liquid traps.
6. An inert-gas (moisture free) system must be provided to purge the transfer lines without the necessity of dumping the residual propellant or disconnecting any system joints.
7. The system components must be reliable, compatible with the oxidizer, and properly serviced (cleared and passivated).
8. The chlorine pentafluoride vents should be ducted together and connected to a vapor scrubber or high vent stack.
9. Sufficient remotely actuated control equipment must be provided to isolate portions of the system during emergencies or components replacement.

The continual observation of an operational system for possible malfunctions can prevent serious propellant spills. The leakage of chlorine pentafluoride is not always apparent because its vapors are colorless. However, a sensitive halogen detector can be used effectively to check the system joints because a small propellant leak yields high local vapor concentrations. Thus, if a small leak is detected, corrective action must be taken as soon as possible.

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5.1.2 Trained Personnel

Properly trained personnel are required to handle chlorine pentafluoride safely. Operating personnel should be thoroughly familiar with the following:

1. The properties of chlorine pentafluoride
2. Operation of the transfer and storage system
3. Toxicity and physiological effects of the propellant
4. Operation and use of safety equipment
5. Fire and spill prevention techniques
6. Fire and spill control measures
7. Disposal and decontamination techniques
8. Local operating procedures and regulations
9. First aid techniques

No person should be allowed to handle chlorine pentafluoride unless thoroughly familiar with the previously listed items and confident that the propellant can be handled safely with the equipment and facilities available. In addition, all operations should be controlled by a procedures checklist, which has been prepared and thoroughly checked by personnel most familiar with the potential problem areas.

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5.2 HAZARD CONTROL

Because the probability of experiencing a propellant leak or spill at some period in its utilization is always present, personnel should be thoroughly educated as to the potential effects and their control. Although detailed hazard classification (cause and effect) and control studies have not been performed with chlorine pentafluoride, the general philosophy used thus far in its handling is identical to that suggested for chlorine trifluoride.

In the event of chlorine pentafluoride spillage or fire, all personnel should report to predesignated safe areas or emergency operating posts (usually positive pressure shelters and/or upwind areas). Immediate evaluation of the hazardous situation is necessary so that appropriate control action can be initiated in the shortest possible time.

The time period between the inception of the hazardous situation and initiation of control action shall be reduced to a minimum. This can be accomplished through proper planning, training, and organization. The following items shall be considered in the administration of the storage and handling areas:

1. Safe areas and evacuation routes shall be pre-established through cognizance of local meteorology conditions.
2. Only authorized personnel shall be allowed to enter storage and handling areas.
3. A minimum of two operating personnel shall wear protective clothing and equipment during propellant handling operations.
4. Periodic drills shall be performed to ensure personnel proficiency during emergency operations.

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5.2.1 Spill Control

A propellant spill can be most efficiently controlled by performing the following steps chronologically:

1. Stop the propellant handling operations.
2. Isolate the propellant tanks from the transfer lines by closing the necessary valves (by remote control if possible).
3. Locate the source of spill.
4. Isolate the affected components by closing the necessary valves.
5. Dispose of the spilled propellant.

The performance of the first four steps listed previously should be automatic and can be performed in a very short time.

The disposition of the spilled propellant should not be too difficult, especially when propellant handling is performed only during satisfactory weather conditions and the first four steps listed previously are quickly executed. The disposition method depends greatly on the quantity of propellant spilled, prevailing weather conditions, location of storage and/or handling area, etc. Therefore, the discussion presented herein will be limited to general criteria which will be applicable to most situations.

Chlorine pentafluoride spills can be best controlled by allowing the propellant to vaporize. In this case, a large amount of propellant would initially vaporize (flash) resulting in the cooling of the residual propellant. Shortly thereafter, steady-state

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vaporization is experienced. It should be noted that a large amount of toxic vapors are generated over a long period of time, thus dictating the need of performing propellant handling operations during satisfactory weather conditions.

As an alternate method, the spilled propellant can be deluged with large quantities of water. In this case, however, the water reacts with the propellant forming hydrofluoric acid and generating large quantities of energy. This method is not recommended for the following reasons:

1. A large quantity of hydrofluoric acid is formed which might be difficult to dispose of subsequently because of water pollution problems.
2. The water might react explosively with the propellant.
3. The energy liberated by the reaction would cause rapid vaporization of the residual propellant which can result in prohibitive vapor concentrations.

After the spill is controlled, the entire area must be thoroughly decontaminated. Decontamination techniques are presented in another section of this document.

5.2.2 Fire Control

Chlorine pentafluoride fires result in the generation of intense heat for a short period of time. Because the propellant fires cannot be extinguished efficiently, if at all, the fire control techniques are based on preventing facility damage. This is accomplished by deluging the area with copious quantities of water. Fog-type water injection nozzles are particularly suitable for this application.

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The reduction or prevention of facility damage resulting from fires can be attained when the following items are considered in the design, fabrication, and operation of the storage area:

1. The facility must be designed as fireproof as possible.
2. The area must be maintained clean, uncluttered, and free from combustible materials.
3. The facility must be equipped with a properly designed water-deluge system, preferably of the fog type.
4. The storage tanks must be diked to limit the potential propellant burning zone.

It is emphasized again that the most important way of preventing facility damage is by preventing chlorine pentafluoride leaks and spills.

5.3 SAFETY EQUIPMENT

The toxic and extremely reactive properties of chlorine pentafluoride dictate the need for adequate safety equipment to protect operating personnel and facilities. It should be recognized that the type of personal safety equipment recommended for a given situation must be consistent with the potential hazard extent, e.g., liquid impingement, vapor inhalation, etc. The category of safety clothing required for a given job must be specified at the time it is assigned. Preferably, written job instructions should be given to ensure adequate communication and agreement between operating and responsible safety personnel.

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5.3.1 Facility Safety Equipment

Equipment for facility protection shall consist of a water deluge system (preferably of the fog type), portable fire extinguishers, and fire hoses. This equipment shall be strategically located and easily accessible. Other facility items to be provided for personnel protection include safety showers, eye wash fountains, and appropriately located first-aid kits.

All operating personnel shall be thoroughly familiar with the location and operation of each piece of safety equipment. The operating condition of the equipment must be verified periodically.

5.3.2 Personal Protective Equipment

All personnel in the chlorine pentafluoride handling and storage areas shall wear flame-retardant clothing at all times. In addition, all personnel performing propellant transfer operations shall wear fully protective personal equipment. If the operations are performed remotely, it is still recommended that at least two operating personnel be fully protected to facilitate proper spill and fire control. A chlorine pentafluoride vapor detector should be used to determine vapor concentration present in the area prior to removal of protective equipment.

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The following items of personal protective equipment, or their equivalent, have been used with satisfactory results:

1. Flame retardant coveralls (worn as an inner garment)
2. Apron (open back) or rocket fuel handler's coverall, 406F-116, DuPont Armalon (choice of outer covering dependent on quantity of propellant involved)
3. Neoprene-type gloves
4. Hood, 406F-116 DuPont Armalon with 0.080-inch methyl methacrylate window
5. Neoprene-type boots
6. Supplied Air Breathing Apparatus (SABA) or airline respirator

This equipment must be maintained clean and in good operating order. A contaminated suit, for example, can become a definite safety hazard.

NOTE: The preceding equipment recommendations are based only on their commercial availability and previous history of use. Safety equipment specifications and limitations should be checked with the manufacturer before use. In general, protective clothing impermeable to fluorine compounds is acceptable, although under certain conditions these materials may react.

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5.4 DECONTAMINATION

Decontamination involves the removal of chlorine pentafluoride, hydrofluoric acid, and other fluorides following a propellant spill or fire, and the deactivation of facility equipment previously exposed to chlorine pentafluoride. Decontamination procedures are employed to protect both personnel and equipment. Personnel performing these operations shall wear the full protective equipment described in the Safety Equipment section.

5.4.1 Area Decontamination

The contaminants remaining from a chlorine pentafluoride spill or fire are hydrofluoric acid, solid fluorides, and in some cases, liquid chlorine pentafluoride. Because these fluorine compounds are corrosive and toxic, they must be removed. This can be accomplished by washing the area with copious quantities of water. The drained water in turn becomes contaminated and must be disposed of as stipulated by local water pollution control regulations.

5.4.2 Equipment Decontamination

The removal of a component from a chlorine pentafluoride system must be preceded by a thorough inert gas (dry) purge to remove any residual propellant. If the removed component is to be reused without service or modification, no further decontamination operations are required; otherwise, the removed component is purged thoroughly with water and dried by purging it with inert gas.

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All components removed from a chlorine pentafluoride system must be labeled clearly, describing the extent of decontamination and operational status.

Any component or system exposed to the atmosphere, water, cleaning solvent, etc. must undergo cleaning and passivation (described previously) before its reuse with chlorine pentafluoride. In addition, as shown in the Materials section, the prolonged contact of chlorine pentafluoride or fluoride films with even small amounts of moisture will result in excessive corrosion of normally compatible metals and potentially hazardous situations.

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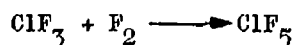
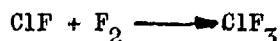
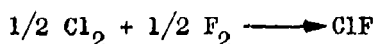
SECTION 6: LOGISTICS

6.1 PRODUCTION

6.1.1 Synthesis Techniques

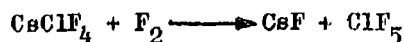
The first synthesis of ClF_5 was accomplished by reaction of a fluorine, chlorine, and nitrogen mixture in glow-discharge apparatus at -80°C (Ref. 1). It was soon found that only chlorine and fluorine or fluorine and chlorine trifluoride were necessary and sufficient for this preparation; however, the glow-discharge technique did not provide adequate amounts for characterization. Rocketdyne investigators also found that ClF_5 could be prepared through a variety of techniques (Ref. 3). Three of these which were used successfully are described below.

Direct combination of chlorine and fluorine at temperatures up to 285°C gives ClF_5 at moderate pressures (500 to 1500 psi). This reaction probably proceeds in a stepwise process in the sequence:



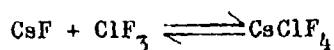
As is apparent from this reaction sequence, ClF_5 can also be prepared from the reaction of ClF_3 with F_2 .

On the laboratory scale, a convenient method for preparing ClF_5 is through the fluorination of an alkali-metal tetrafluorochlorate, MClF_4 . Cesium tetrafluorochlorate has been preferred because of favorable equilibrium and handling properties when working on a laboratory scale.



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This reaction has several advantages. Only one condensable gaseous product is produced, making purification relatively simple. The CsClF_4 can also be regenerated with ClF_3 by the reaction:



In addition to these initial techniques, electrochemical synthesis of ClF_5 has been demonstrated (Ref. 58 and 59). During these studies, both the HF-NaF-ClF_3 system and the HF-NaF-Cl_2 (gas) system were used in the preparation of ClF_5 .

Although detailed information on the present production techniques of various facilities is restricted, the methods primarily utilized are the direct combination of chlorine or chlorine trifluoride with fluorine (and modifications thereof). The development and operation of an initial pilot plant, employing a 12 lb/day continuous-flow process, has been described by Rocketdyne (Ref. 6).

6.1.2 Availability

Thus far, three different production facilities, Aerojet-General Corporation, Allied Chemical Corporation, and Rocketdyne, are presently capable of supplying ClF_5 in quantities of several thousand pounds. It is assumed that all three facilities will continue to supply ClF_5 in the future at rates equivalent to the demands. However, it is apparent that future requirements will necessitate expansion of all present facilities.

6.1.3 Cost

The initial cost (~\$200 to \$400 per pound) of high-purity ClF_5 reflected the cost of process research and development. During subsequent procurements of larger quantities, the cost was reduced \$20 to \$30 per pound. An appraisal of the production process indicates that the eventually obtainable cost of ClF_5

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will be comparable to that of liquid fluorine at the same usage rates. (Potential cost of liquid fluorine at high usage rates has been quoted as ~\$1.00 per pound.) The reduction of the current cost to that projected level will be a direct function of the demand. Cost quotations for a particular quantity over a defined period should be obtained from the suppliers.

6.1.4 Propellant Specification

Although there is no present government specification for ClF_5 , a formal specification is being prepared by the Air Force Rocket Propulsion Laboratory, Edwards, California. This specification will designate a propellant grade purity of 99+ weight percent ClF_5 for procurement purposes.

6.1.5 Chemical Analysis

The complete chemical analysis of ClF_5 is presently conducted at Rocketdyne with a combination of two techniques: vapor phase chromatography and infrared spectrophotometry. The combination of these techniques assays for F_2 , HF , ClF , CF_4 , SiF_4 , SF_6 , FClO_3 , Cl_2 , FClO_2 , ClO_2 , and ClF_3 present in ClF_5 . The procedure is described in the following paragraphs.

6.1.5.1 Chromatographic Analysis. A 2- to 5-gram liquid sample of ClF_5 is completely vaporized into a large-volume cylinder attached to the manifold of a custom-built chromatograph (R.f. 60) that has been thoroughly passivated with ClF_3 . Sufficient vapor is admitted into the chromatograph to develop 400 to 800 mm Hg pressure in a 5- or 10-cc sample loop.

The vapor is separated on a Halocarbon gel column of 50 percent 4-11v Halocarbon oil on 40-60 mesh low-density Kel-F molding powder packed in 30 feet of 3/16-in diameter stainless-steel

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tubing. The components are eluted from the column in the following order: ($O_2 + N_2 + F_2 + CF_4 + SiF_4$), SF_6 , ClF , $FClO_3$, Cl_2 , ($FClO_2 + ClO_2$), ClF_3 , and ClF_5 . From the chromatogram, the mole percentage of the individual components, the sum of $F_2 + CF_4 + SiF_4$ and the sum of $FClO_2 + ClO_2$ can be calculated.

6.1.5.2 Infrared Analysis. Part of the originally vaporized sample is introduced, usually at a pressure of 75 mm Hg, into a 5-centimeter Monel gas cell fitted with silver chloride windows. Using the infrared absorption curve, the mole percentages of CF_4 , $FClO_2$, and SiF_4 can be calculated from the absorption bands at 7.75, 7.95, and 9.7 microns, respectively.

A third portion of the sample is introduced into a 7.5-centimeter Monel cell fitted with calcium fluoride windows to a pressure of 500 to 750 mm Hg. The spectrum from 2700 to 2500 millimicrons is scanned. The absorption peak of HF at 2575 millimicrons is measured and the mole percentage of HF is calculated using a Beers-Lambert calibration plot of anhydrous HF vapor.

6.1.5.3 Typical Analysis. A typical chemical analysis of the material produced in the Rocketdyne ClF_5 production plant is presented in Table 7.

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TABLE 7

TYPICAL ASSAY OF CHLORINE PENTAFLUORIDE

LOG # 6-4-300

CERTIFICATE OF ANALYSIS

FLUORIDYNE

CONSIGNEE _____

CYLINDER NO. W 1481

DATE 5/3/66

LOT NO. 42

WEIGHT PERCENT

ASSAY (MINIMUM)	99.4+
HYDROGEN FLUORIDE	0.3
CHLORINE MONOFLUORIDE	0.03
CHLORINE	< 0.02
CHLORINE DIOXIDE	0.09
CHLORINE TRIFLUORIDE	0.06
PERCHLORYL FLUORIDE	< 0.02
NON CONDENSABLES (as F ₂)	0.04

GROSS 410 LBS.

TARE 195 LBS.

QUANTITY 215 LBS.

Q.C. NO. _____

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6.2 TRANSPORTATION

Shipment of chlorine pentafluoride by common carrier is authorized by the Interstate Commerce Commission (ICC) which classifies ClF_5 as a "Corrosive Liquid." It also has an "NOS" (not otherwise specified) designation. In transit, cylinders are marked as Compound A, Corrosive Liquid, and Flammable Liquid Only and must be affixed with an ICC-approved WHITE label. Highway vehicles carrying 2500 pounds or more of ClF_5 must be identified with "Dangerous" placards in letters at least 3 inches high on a contrasting background.

Chlorine pentafluoride has been shipped under its own vapor pressure in a variety of different sized cylinders in quantities ranging from a few grams to 165 pounds (ICC 3AA 2400) per cylinder; these cylinders are equipped with one shutoff valve. Eventually, it is anticipated that larger cylinders, equipped with both vapor and liquid shutoff valves, will be approved and utilized. At present, the ICC specifications applicable to the shipping cylinders have been authorized under ICC special permits obtained in accordance with Agent T. C. George's Tariff No. 15, "Interstate Commerce Commission Regulations for Transportation of Explosives and Other Dangerous Articles by Land and Water in Rail Freight Service and By Motor Vehicle (Highway and Water)." These special permits are granted to the individual suppliers by the ICC Bureau of Operations and Compliance, Washington, D. C., 20423. The applicable shipping cylinder specifications relative to each shipment should be obtained from the supplier prior to delivery.

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6.3 STORAGE

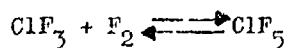
6.3.1 Storability Test Analysis

There have been several studies conducted to establish the storability of ClF_5 under various conditions. These tests and their results are characterized in Table 8.

6.3.2 Thermal Stability and Equilibrium Analysis

In addition to basic thermal stability data developed during the high-temperature storability tests, materials compatibility tests, and physical property measurements, the thermal stability characterization of ClF_5 was attempted through the use of the standard JANAF thermal stability apparatus (Ref. 6). Each test, which involved heating of the ClF_5 sample at a rate of 20 F/minute, was terminated at approximately 680 F by the rupture of a 5300-psi burst diaphragm in the apparatus. Although there was no detectable evidence of decomposition of ClF_5 (by ΔT measurements between the heating bath temperature and sample temperature), during three tests, it should be noted that small effects are not detected by this relatively crude apparatus. The data obtained during the thermal equilibrium studies subsequently discussed, provide a more accurate knowledge of thermal stability.

Early in the synthesis of ClF_5 , it is apparent that dissociation of ClF_5 occurs at elevated temperatures. Accordingly, a study was undertaken (Ref. 8) to ascertain the nature of the dissociation and measure the temperature dependence of the equilibrium constant. Studies were conducted in the range 210 to 270 C (410 to 518 F). For the reaction



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TABLE 8
CHLORINE PENTAFLUORIDE STORABILITY TESTS

Container		Test Conditions				Storage Time	Results (d)	Reference Number
Material	Volume	Ullage, volume percent	Temperature (a)	Sample Purity, (b) weight percent ClF ₅	Conditioning (c)			
321 Stainless Steel	7 cc	50	Ambient	99+	Net	6 weeks	1	6
						12 weeks	2	6
						18 weeks	3	6
						24 weeks	3	6
						32 weeks	3	6
	206.5 cc	21.5 to 40			Dry	15 months	1	9
	17 cc	60	160 F			30 days	1	6
	8 gallon	Unspecified (varied)	Ambient			7 months	1	6
	1.5 gallon	Unspecified (varied)	Ambient			15 months	1	6
347 (three containers)	1 quart	5	104 F		Not specified	10 days	1	13
317 Stainless Steel	1 quart	2	160 F			454 days	3	61
(Continuation of above tests under new conditions)								
Stainless Steel (type unspecified)	Unspecified	Unspecified	160 F	90+		60 days	1	4
304 Stainless Steel	Unspecified	Unspecified	167 F	99+		84 days	1	13
6061 Aluminum (76)	7 cc	50	Ambient		Net	6 weeks	1	6
						12 weeks	2	6
						18 weeks	2	6
						24 weeks	3	6
						32 weeks	3	6
	231.5 cc	31 to 30			Dry	15 months	1	9
	17 cc	60	160 F			30 days	1	6
7052-O Aluminum	7 cc	50	Ambient		Net	6 weeks	1	6

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TABLE 8
(Concluded)

Container		Test Conditions					Storage Time	Results (d)	Reference Number
Material	Volume	Ullage, volume percent	Temperature (a)	Sample Purity, weight percent CIF ₅ (b)	Conditioning (c)				
5052-0 Aluminum	7 cc	50	Ambient	99+	Wet	12 weeks	2	6	
						18 weeks	2		
						24 weeks	3		
						32 weeks	3		
						30 days	1		
Copper (commercial)	7 cc	50	Ambient		Wet	6 weeks	1		
						12 weeks	2		
						18 weeks	2		
						24 weeks	3		
						32 weeks	3		
Monel 400	215 cc	24 to 41	160 F		Dry	15 months	1	9	
	17 cc	60	Ambient		Dry	30 days	1		
	218 cc	28.5 to 48	Ambient		Dry	15 months	1		

(a) Temperature represents average storage temperature; ambient indicates a prevailing temperature range (50 to 100 F)

(b) Sample purity represents analysis at initiation of test

(c) Conditioning indicates presence of moisture at loading

(d) Results are indicated by:

1. No evidence of propellant breakdown
2. Trace propellant breakdown
3. Significant propellant breakdown

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the equilibrium constant, K_p , is best expressed over this range by the equation

$$\log K (\text{atm}^{-1}) = \frac{3984}{T(K)} - 9.25$$

It was significant that no dissociation of ClF_5 was observed at 165 C (329 F), well above the critical temperature of 143 ± 0.5 C (289.4 ± 0.9 F). Either the noted equilibrium constant, determined over the higher temperature range, cannot be extrapolated to lower temperatures or, more probably, the decomposition rate at 165 C (329 F) is extremely slow.

6.3.3 General Storage Requirements

From the available storability and thermal stability data it is obvious that ClF_5 may be stored safely under prevailing ambient temperatures for long periods of time provided the storage system is fabricated from compatible materials, properly cleaned and passivated, and kept free of moisture (and/or other contamination) and excessive heat. Although storage data have been limited to periods of < 2 years, it is anticipated (based on the apparent inherent stability of ClF_5 and extrapolated materials compatibility data) that ClF_5 may be stored for periods of several years without adverse effects on either the propellant or material. Future analyses of longer-term storage tests currently in progress will provide additional data relative to potential time limitations.

The shipping containers in which the propellant is received are satisfactory for storage. However, frequent inspection of the valves and fittings is mandatory because of the corrosive nature of the propellant. These cylinders must be located in a designated safe storage area and positioned so that they are secured against rolling or being inadvertently tipped over. This can be accomplished by placing them in cradles or by securing them in a vertical position with chains and individual cylinder supports.

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Storage of ClF_5 in containers other than the shipping cylinders can be effected in tanks fabricated of compatible materials (i.e., stainless steels, aluminum alloys, Monels, etc.), provided they are properly designed, fabricated, cleaned, and passivated prior to use.

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6.4 HANDLING

Chlorine pentafluoride handling operations, as described herein, include generalized recommended handling techniques for the unloading of the shipping cylinders, loading of storage tanks, venting, and disposal operations. All other handling operations are specific to particular systems and cannot be treated with generalized criteria. Personnel performing handling operations must wear fully protective equipment described in the SAFETY EQUIPMENT section. Another activity closely associated with the above functions is the handling of the shipping cylinders. The shipping cylinders can be handled safely without the need of fully protective equipment.

6.4.1 Handling of Shipping Cylinders

The shipping cylinders must be handled with extreme care. The cylinder shutoff valve(s) cap(s) must be installed at all times during cylinder handling operations.

The cylinders may be transferred by means of any piece of equipment capable of handling them safely. While in transfer, the cylinders must be firmly secured.

Storage of the shipping cylinders should be restricted to those areas specifically designated for this purpose. The condition of each cylinder (full, empty, contaminated, etc.) must be marked clearly.

6.4.2 Transfer of Chlorine Pentafluoride from Shipping Cylinders

Chlorine pentafluoride can be discharged from its storage container either by its own vapor pressure, by pressurizing the container with dry nitrogen or helium, or by connecting a

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transfer pump in the container discharge line. Inert gas pressurization is used almost exclusively at the present time for unloading large cylinders because this technique is extremely reliable. The transfer pump technique is practical when large quantities of the propellant must be transferred in a relatively short period of time. Vapor pressure unloading is used primarily in the transfer of small quantities and is impractical in the handling of large propellant quantities.

The propellant transfer system must be chemically compatible with the propellant, leakproof, and in excellent operating order. The complete system must also be passivated immediately prior to the commencement of complete flow conditions.

In preparing for a transfer operation, all personnel not directly concerned with the operation shall evacuate the hazard area. Appropriate warning lights and signs shall be displayed to keep out unauthorized personnel.

Personnel performing the transfer operation shall wear the fully protective equipment described in the Safety Equipment section. If the operations are performed remotely, at least two operating personnel should be fully dressed to facilitate proper spill and fire control. Sufficient safety equipment should be available for all personnel allowed to remain in the hazard area.

Supervisory and emergency support personnel shall be notified prior to executing any hazardous operation in the storage area.

The propellant transfer procedures are dependent upon numerous factors such as transfer system design, type of propellant container, training of operating personnel, prevailing weather conditions, etc. Establishing proper operating procedures for each specific situation in a single document is not feasible.

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Therefore, the procedures presented in the following paragraphs are general in nature. The transfer system schematics presented (Fig. 11 through 13) are not finalized designs; they are provided only to facilitate the explanation of typical procedures.

6.4.2.1 Transfer From Single-Opening Containers. The currently available ClF_5 cylinders are single-opening containers in which only one opening is available to perform the propellant transfer operation. The opening is sealed by a compatible shutoff valve, which in turn is protected by a gas-tight cap. These cylinders are not equipped with dip tubes and as such should be avoided because they present additional propellant transfer complexity.

The propellant can be transferred from these cylinders by prepressurizing the cylinders with dry nitrogen prior to the transfer, or by allowing the propellant to flow under its own vapor pressure. The vapor transfer technique is inefficient, and in some cases, impossible, unless the collecting tank is cooled. Therefore, the prepressurization technique is recommended for most operations involving single-opening cylinders.

The prepressurization of the cylinder with dry nitrogen can be accomplished as follows:

1. Remove the protective cap from the cylinder shutoff valve.
2. Connect a clean, regulated, dry-nitrogen supply line to the shutoff valve of the cylinder.

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3. Regulate the pressure supply to the desired value.
The regulated pressure level determines the rate of propellant transfer. A value of approximately 100 psig is usually adequate. The pressure should never exceed 10 psig less than the cylinder design pressure.
4. Open the pressure supply shutoff valve.
5. Slowly open the cylinder shutoff valve.
6. When the cylinder pressure equalizes the regulated source pressure, close the supply and cylinder shutoff valves.

NOTE: Two basic techniques can be used to determine when pressure equalization is attained. First, the noise generated by the gas flow through the pressurizing line ceases. Second, the regulated pressure gage registers the regulated pressure value prior to gas flow.

7. Bleed the trapped gas between the two shutoff valves by opening the transfer-line bleed valve.
8. Disconnect the pressurizing line from the cylinder shutoff valve.
9. Cap the opened connections to prevent contamination.

The transfer of ClF_5 from the pressurized cylinder to the storage tank (Fig. 11) can be performed as follows:

1. Turn the cylinder upside down and place it in a transfer cradle. The cylinder must be properly secured and care must be exercised to prevent damage to the cylinder shutoff valve.
2. Connect the cylinder shutoff valve to the propellant transfer system as shown in Fig. 11.
3. Close all system valves except valve No. 6 which must be maintained open. The purpose of valve No. 6 is to prevent the continuous escape of ClF_5 in case of

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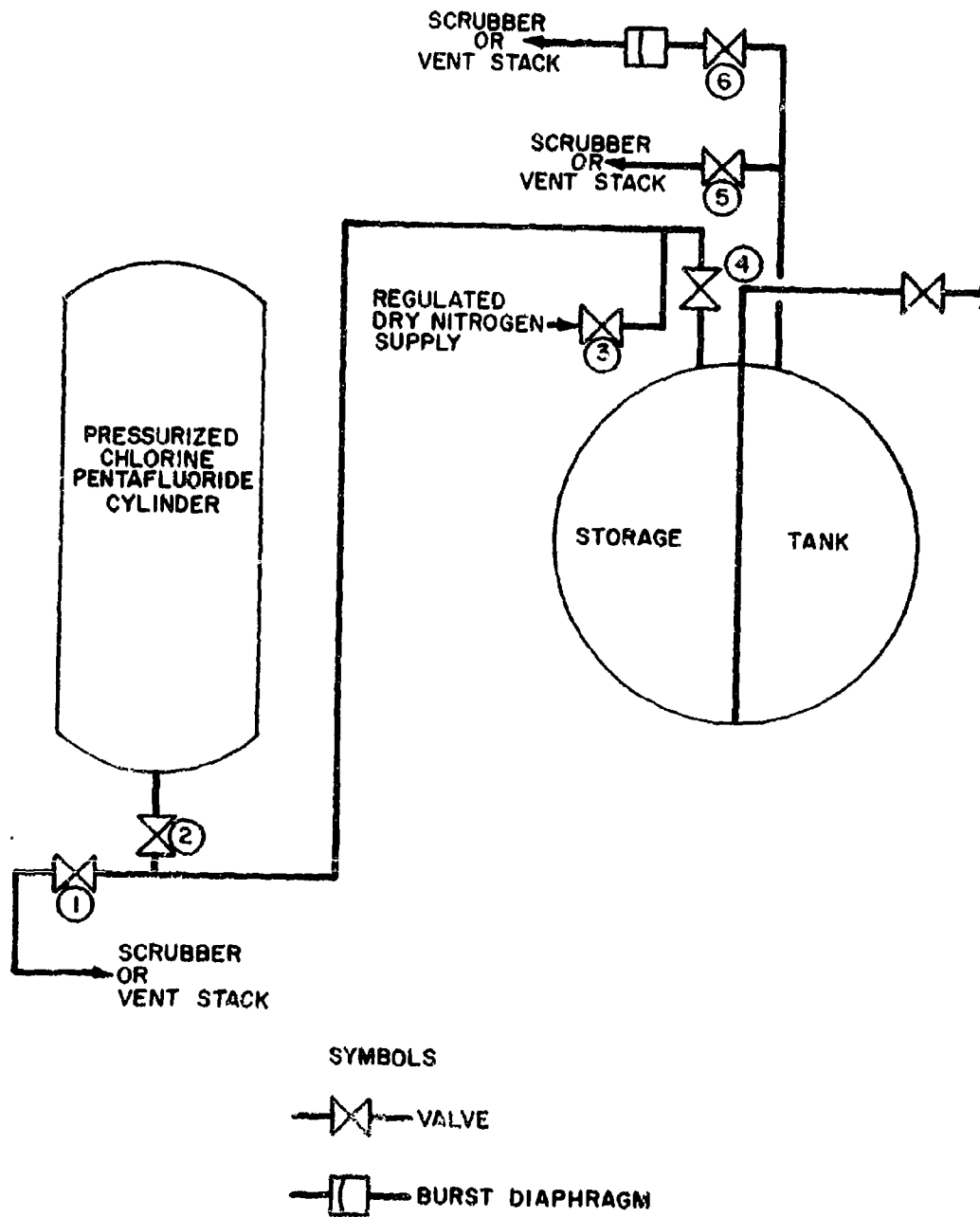


Figure 11. Transfer of Chlorine Pentafluoride From Pressurized Single-Opening Cylinders

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burst-diaphragm failure and to facilitate the removal of the burst diaphragm whenever required.

4. Purge the propellant transfer line to remove any residual trapped water vapor. This is accomplished by opening valves No. 3 and 1. When the purge operation is completed (approximately 3 minutes), close valves No. 3 and 1.
5. Open valve No. 2 slowly and check for leaks. If a leak develops, close the valve, open valve No. 1, and take the action necessary to stop the leak. (Valve No. 1 must be closed and valve No. 2 opened before proceeding with Step 6.)
6. Open valve No. 4.
7. After valves No. 2 and 4 have been opened, the propellant flows from the cylinder into the storage tank until the liquid in the cylinder is depleted or the pressure in the two containers equalizes. If the pressure in the two containers equalizes, close valve No. 4, and open valve No. 5 momentarily to depressurize the storage tank. The flow can be resumed by reopening valve No. 4.
8. When the desired quantity or all of the available propellant has been transferred, close valves No. 2 and 4.

NOTE. There are several devices that can be used to detect the completion of the propellant transfer operation. Combinations of two or more devices are usually required to provide the desired flexibility of the transfer system. Some of these devices are:

- a. A flowmeter installed in the transfer line
 - b. A scale or other weight-sensing device attached to the container being unloaded
 - c. A calibrated level indicator mounted on the storage tank
9. Purge the transfer line thoroughly by opening valves No. 1 and 3. When the purging operation is completed (approximately 3 to 5 minutes), close valves No. 3 and 1.

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10. Depressurize the storage tank to a safe, positive pressure level by opening valve No. 5 momentarily.
11. Disconnect the cylinder shutoff valve from the transfer system and cap the opened components.
12. Turn the cylinder to the upright position, mark it adequately, and dispose of it according to operating procedures.
13. Notify all personnel concerned that the transfer operation is completed and the area is clear.

6.4.2.2 Transfer From Double-Opening Containers. At the present time, there are no double-opening shipping containers in ClF_5 service; however, the eventual use of cylinders similar to the 1-ton ClF_3 cylinders is assured. The operation of these cylinders in ClF_5 service will be identical. The 1-ton ClF_3 shipping cylinder is a double-opening container and is equipped with two shutoff valves. During transfer operations, one opening can be used to pressurize or vent the container and the other opening to discharge the propellant.

The propellant can be discharged from the shipping container either by pressurizing the container with dry nitrogen or by connecting a transfer pump in the product discharge line. Although the pressurization unloading technique has been used nearly exclusively in the past, procedures for both transfer techniques are discussed in detail below.

6.4.2.1 Pressurization Unloading. As mentioned previously, the transfer of liquid ClF_5 from shipping containers can be performed reliably by pressurizing the containers with dry nitrogen. The following procedure is basically applicable to the transfer of the propellant from double-opening containers into a storage tank using gas pressurization.

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1. Place the 1-ton cylinder in the horizontal position with the shutoff valves aligned with the vertical centerline axis.
2. Remove the protective cap from the cylinder and connect the cylinder shutoff valves to the transfer system as shown in Fig. 12. When the cylinder is positioned according to Step 1, above, the upper shutoff valve is connected to the regulated pressure supply, and the lower shutoff valve is connected to the transfer line.
3. Ensure that all system valves are closed except valve No. 9 which must be maintained open. The purpose of valve No. 9 is to prevent the continuous escape of ClF_5 in case of burst-diaphragm failure and to facilitate the replacement of the burst diaphragm whenever required.
4. Set the dry nitrogen regulator to the desired pressure level. This pressure level determines the propellant discharge flow. A value of ~ 100 psig is usually adequate. The pressure should never exceed 10 psig less than the container design pressure.
5. Purge the propellant lines to remove residual trapped water vapor. This can be accomplished as follows:
 - a. Open valves No. 4 and 7, and purge for approximately 3 minutes
 - b. Close valve No. 7 and 4
 - c. Open valves No. 3 and 2, and purge for approximately 3 minutes
 - d. Close valves No. 2 and 3
6. Open valve No. 5 and check for leaks. If a leak develops, close the valve, open valve No. 4, and take the necessary action to stop the leak. (Valve No. 4 must be closed and valve No. 5 opened before proceeding with Step 7.)

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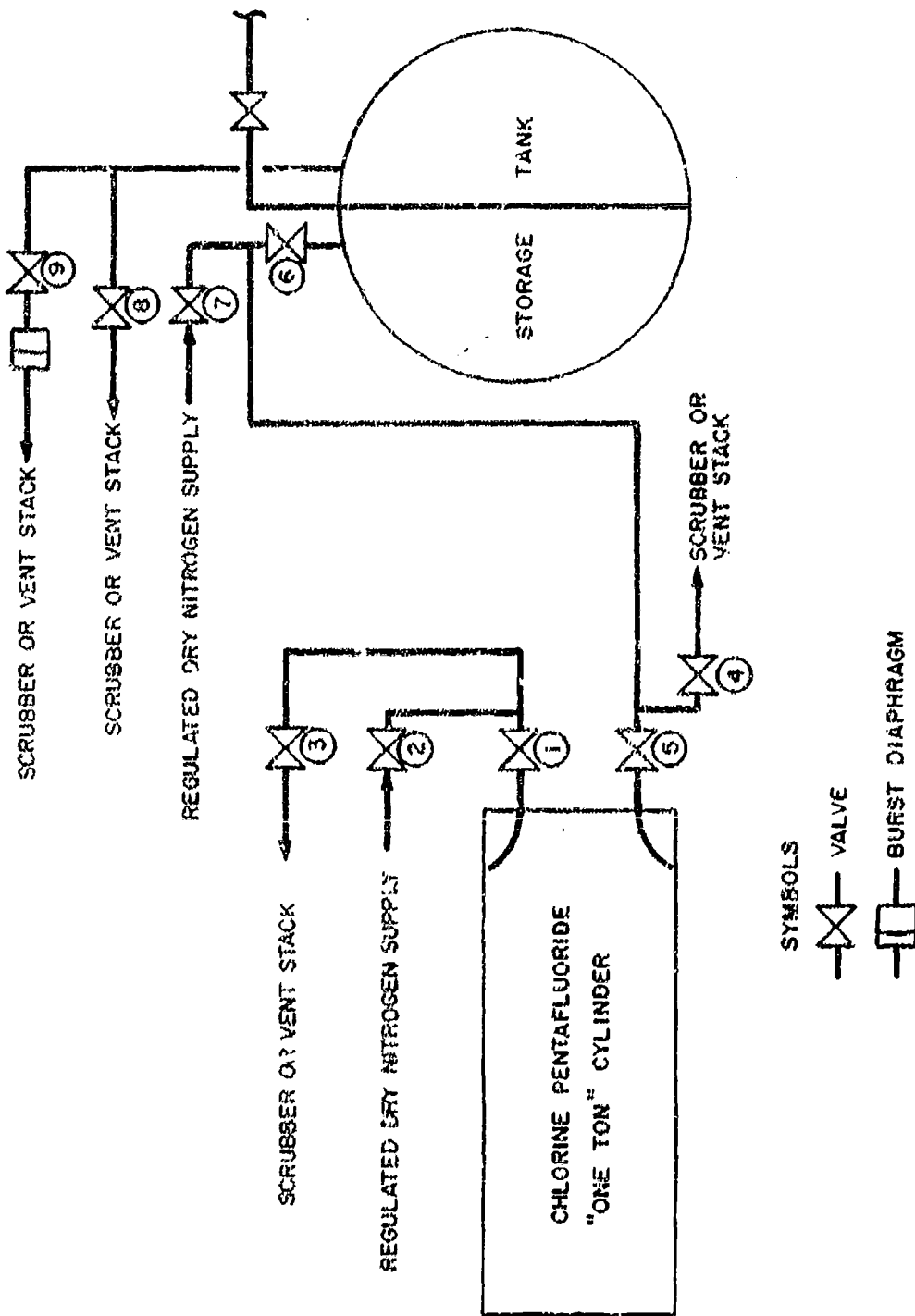


Figure 12. The Transfer of Chlorine Pentafluoride from Double-Opening Containers Using Gas Pressurization

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7. Open valve No. 6
8. Establish the propellant flow by pressurizing the ClF₅ cylinder. This is accomplished by opening valves No. 1 and 2. A propellant flow is experienced until the liquid in the shipping container is depleted or the pressure in the two containers equalizes. If the pressure in the two containers equalizes, close valves No. 2 and 6, and open valve No. 8 momentarily. The flow can be resumed by reopening valves No. 6 and 2.
9. When the desired quantity or all of the available propellant has been transferred, close valves No. 2 and 5.

NOTE: There are several devices which can be used to detect the completion of the propellant transfer operation. Combinations of two or more devices are usually required to provide the desired transfer system flexibility. Some of these devices are:

- a. A flowmeter installed in the transfer line
 - b. A scale or other weight-sensing device attached to the container being unloaded
 - c. A calibrated level indicator mounted on the storage container
10. Depressurize the shipping container by opening valve No. 3. When the container is depressurized, close valves No. 1 and 3.
 11. Close valve No. 6 and purge the transfer line by opening valves No. 4 and 7. When the transfer line is properly purged (usually 3 to 5 minutes at a pressure level of approximately 50 psig), close valves No. 7 and 4.
 12. Depressurize the storage container by opening valve No. 8 for a short period of time.
 13. Disconnect the shipping cylinder shutoff valves from the transfer system and cap the opened components.
 14. Mark and dispose of the shipping container according to operating procedures.

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15. Notify all personnel concerned that the transfer operation is completed and the area clear.

6.4.2.2.2

Transfer Pump Unloading. As mentioned previously, pump unloading is a potential alternate method of transferring ClF_5 from the shipping cylinders into storage tanks. This technique is highly applicable when large quantities of the propellant must be transferred in a relatively short period of time.

The following procedure is basically applicable to the potential transfer of ClF_5 from a "1-ton" type shipping cylinder into a storage tank by means of a transfer pump:

1. Place the shipping cylinder in a horizontal position with the shutoff valves aligned with the vertical centerline axis.
2. Remove the cylinder protective cap and connect the cylinder shutoff valves to the transfer system as shown in Fig. 13. With the cylinder positioned according to Step 1, above, the upper shutoff valve is connected to the vapor-return line and the lower shutoff valve is connected to the transfer line.
3. Ensure that all system valves are closed, except valves No. 11 and 7, which must be opened. The objective of valve No. 11 is to prevent the continuous escape of ClF_5 in case of burst-diaphragm failure and to facilitate the removal of the burst diaphragm whenever required. Valve No. 7 prevents pump damage caused by overpressures resulting from the possible vaporization and expansion of trapped propellant in the pump.

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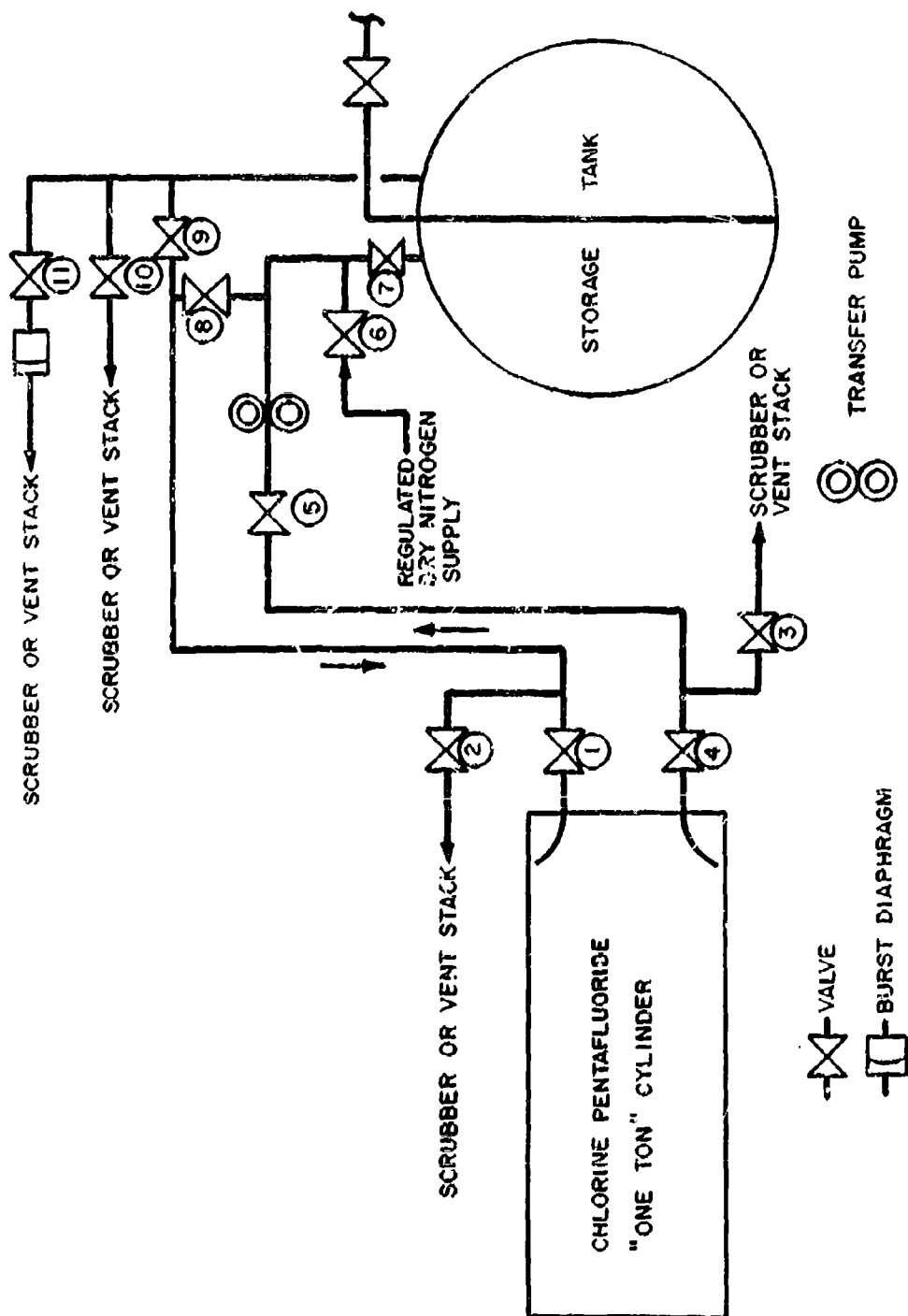


Figure 13. The Transfer of Chlorine Pentafluoride from Double-Opening Containers Using a Pump

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4. Set the dry nitrogen regulator to the desired pressure level. A value ranging from 60 to 70 psig should be adequate.
5. Purge the propellant lines to remove trapped water vapor. This can be accomplished as follows:
 - a. Close valve No. 7.
 - b. Open valves No. 3, 5, and 6, and purge for approximately 3 minutes.
 - c. Close valves No. 6, 5, and 3.
 - d. Open valves No. 2, 8, and 6, and purge for approximately 3 minutes.
 - e. Close valves No. 6, 8, and 2.
 - f. Open valve No. 7.
6. Pressurize the ClF_3 cylinder to permit the proper priming of the transfer pump, if required. This is accomplished as follows:
 - a. Close valve No. 7.
 - b. Open valves No. 1, 8, and 6.
 - c. When the gas flow stops, close valves No. 6, 8, and 1.
 - d. Open valve No. 7.
7. Open valve No. 4 and check for leaks. If a leak develops, close the valve, open valve No. 3, and take the necessary action to stop the leak. (Valve No. 3 must be closed and valve No. 4 opened before proceeding with Step 8.)
8. Open valve No. 5 and check for leaks. If a leak develops, close valves No. 5 and 7, open valves No. 8 and 2, and take the action necessary to

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stop the leak. (Valves No. 2 and 8 must be closed and valves No. 5 and 7 opened before proceeding with Step 9.)

9. Open valve No. 1.
10. Start the transfer pump and open valve No. 9.
A closed-loop pump transfer operation is thus established.
11. When the desired quantity or all of the available propellant have been transferred, stop the transfer pump and close valve No. 7.

NOTE: There are several devices which can be used to detect the completion of the propellant transfer operation. Combinations of two or more devices are usually required to provide the desired transfer system flexibility. Some of these devices are:

- a. A flowmeter installed in the transfer line
 - b. A scale or other weight-sensing device attached to the container being unloaded
 - c. A calibrated level indicator mounted on the storage tank
12. Close valve No. 9 and depressurize the shipping cylinder by opening valve No. 2. When the container is depressurized, close valve No. 2.
 13. Depressurize the storage tank by opening valve No. 10 for a short period of time.
 14. Purge the propellant transfer line as follows:
 - a. Open valve No. 6 for about 2 to 3 minutes or until the gas flow stops.
 - b. Close valve No. 6.
 - c. Close valve No. 4.
 - d. Open valves No. 3 and 6, and purge the line for approximately 3 to 5 minutes.
 - e. Close valves No. 6, 5, and 3.

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15. Depressurize the shipping cylinder by opening valve No. 2. When the cylinder is depressurized, close valves No. 1 and 2.
16. Purge the vapor-return line as follows:
 - a. Open valves No. 2, 8, and 6, and purge for approximately 3 to 5 minutes.
 - b. Close valves No. 6, 8, and 2.
17. Open valve No. 7.
18. Disconnect the shipping cylinder shutoff valves from the transfer system and cap the opened components.
19. Mark and dispose of the shipping cylinder according to operating procedures.
20. Notify all personnel concerned that the transfer operation is completed and the area clear.

6.4.3

Venting

The frequent depressurization of ClF_5 containers is necessary during handling and test operations. In this operation, a considerable amount of vapor is released which must be handled safely. Two basic methods can be used for handling the propellant vapor. These methods are:

1. The transfer system vent lines are connected to a scrubber system which removes the propellant vapor from the vented gases. Many types of scrubbers and solutions for absorbing the propellant can be used.

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2. The transfer system vent lines are connected to a vent stack which discharges the vented gases at least 60 feet above the highest working point in the area. A low-pressure nitrogen purge can be installed in the stack to further dilute the vented propellant vapor before being discharged into the atmosphere.

Chlorine pentafluoride containers should be vented only under controlled conditions. These conditions are dependent upon area location, weather conditions, etc.

6.4.4

Disposal

Disposal involves the controlled release of ClF_5 from a shipping or storage container into a system capable of disposing of the propellant safely. Military regulations, at the present time limit the disposal of similar propellants to a maximum of 1000 pounds for any one disposal operation.

The following items are essential for the proper selection and safe operation of the ClF_5 disposal area:

1. The disposal area shall be adequately isolated.
2. The disposal area shall be clear of trees, weeds, brush, and other combustibles.
3. The area must be provided with adequate facility safety equipment (refer to Safety Equipment section).
4. One person shall never be allowed to work in the disposal area alone.

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5. The personal safety equipment which was described in the Safety Equipment section must be worn during disposal operations.
6. All personnel not participating in the disposal operation shall evacuate the area.
7. Disposal operations shall be performed only under controlled conditions. These conditions are dependent upon area location, weather conditions, etc.

The following methods can be employed to dispose of ClF_5 :

1. The slow release of the propellant through a high vent stack. The outlet should be at least 60 feet above the disposal area. A dry nitrogen purge must be installed in the stack to dilute the propellant vapor before being exhausted into the atmosphere.
2. The controlled burning of ClF_5 by the use of a fuel such as alcohol or kerosene. This is accomplished by placing a quantity of fuel approximately equal to the quantity of ClF_5 to be disposed of into a burn basin. The oxidizer is then fed slowly into the burn basin and allowed to react with the fuel. For this operation, the ClF_5 supply tank should be located at least 50 feet from the burn basin.

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<p>This interim handbook is a compilation of the engineering properties and handling characteristics of chlorine pentafluoride. The handbook includes data and information on chlorine pentafluoride physico-chemical properties, mixtures, materials compatibility, materials preparation and control, safety equipment, decontamination, production, propellant analysis, transportation, storage, and handling. (C)</p>		

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